STANDARD OPERATING PROCEDURE

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Baseline Analytical Data Validation	**ER-SOP-15.17**	**0**	**12/03/99**
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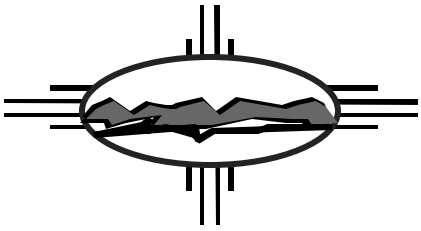

ER PROJECT

LOS ALAMOS NATIONAL LABORATORY

Baseline Analytical Data Validation

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Baseline Analytical Data Validation

NOTE: Environmental Restoration (ER) Project personnel may produce paper copies of this procedure printed from the controlled-document electronic file located at http://erinternal.lanl.gov/documents/Procedures/sops.htm. However, it is their responsibility to ensure that they are trained to and utilizing the current version of this procedure. The Quality Program Project Leader (QPPL) may be contacted if text is unclear.

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the process for performing a baseline validation of analytical data to ensure data quality.

Note: Baseline validation is not to be misinterpreted as a quality-assurance check on contract analytical laboratory (hereafter referred to as the "contract laboratory") performance or systems or a focused validation related to data use.

2.0 TRAINING

All data validators who implement this SOP shall possess a minimum of a Bachelors degree in chemistry and two years experience in generating analytical data in an environmental analytical laboratory, or two years of data validation experience. New validators shall work under the direct supervision of an experienced ER Project validator. The work of new validators shall be reviewed and signed by an experienced ER Project validator until ten data packages for each analytical suite have been satisfactorily validated. ER Project validators shall have a demonstrated familiarity with the U.S. Environmental Protection Agency (EPA) national functional guidelines for data review. All data validators must document that they have read and understand this SOP and completed all applicable training assignments in accordance with QP-2.2.

3.0 DEFINITIONS

3.1 *Analyte* — The element, nuclide, or ion an analysis seeks to determine; the element of interest.

3.2 *Client data package* — The hard copy deliverable for each sample delivery group produced by the contract analytical laboratory in accordance with the statement of work (SOW) for analytical services (RFP No. 9-XS1-Q4257).

3.3 *Contract analytical laboratory* — An analytical laboratory under contract to the University of California to perform analysis of samples for work performed at the Laboratory.

3.4 *Decision level concentration (DLC)* — The concentration at which a 5% probability exists of reporting a false positive for a sample that contains no analytes.

3.5 *Estimated detection limit (EDL)* — The detection limit required by the Laboratory statement of work (SOW) for analytical services (RFP No. 9-XS1-Q4257). The Laboratory value reflect the contract-required detection limits (CRDLs) of the Contract Laboratory Program (CLP) methods.

3.6 *Estimated quantitation limit (EQL)* — The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operation conditions. The low point on a calibration curve should reflect this quantitation limit. See the SOW for analytical services (RFP No. 9-XS1-Q4257) for a more complete definition.

3.7 *Instrument detection limit (IDL)* — The lowest concentration that can be reliably distinguished from the noise level on a particular instrument used in a particular method. For the ER Project, the value is determined by calculating three standard deviations of the results obtained from the analysis of a standard solution (each analyte in reagent water) at a concentration of three to five times the estimated IDL on three nonconsecutive days—with seven consecutive measurements per day.

3.8 *Laboratory qualifier (or Laboratory flag)* — Codes applied to the data by the contract analytical laboratory to indicate data quality concerns. These flags are applied using the Environmental protection Agency (EPA) contract laboratory program (CLP) guidelines.

3.9 *LANL qualifier* — Codes applied to the sample data by data validators who are independent of the contract laboratory which performed the sample analysis and are conducting the analysis as required by SW-846, Risk Assessment Guidance for Superfund (RAGS) and all other data-validation guidance documents. Data qualifiers identify the information bias of sample data (in lieu of a focused validation) needed to determine the limitations on data usability. See Attachment A for the complete list and descriptions of the qualifiers.

3.10 *LANL reason code* — Codes applied to the sample data by data validators who are independent of the contract laboratory which performed the sample analysis. Reason codes provide an in-depth and analysis-specific explanation for applying the qualifier with some description of the potential impact on the data use. See Attachment A for the complete list and descriptions of the reason codes.

3.11 *Method detection limit (MDL)* — The minimum concentration of a substance the can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The MDL is determined from analysis of samples of a given matrix type that contain the analyte after subjecting the sample to the usual preparation and analyses.

3.12 *Minimum detectable activity (MDA)* — The following equation shall be used to calculate the MDA unless otherwise noted or approved by the Laboratory:

$$MDA = \frac{4.65(BKG)^{0.5} + 2.71}{2.22 \times EFF \times V \times T_S \times Y}$$

where BKG = the total background counts,
EFF = the fraction detector efficiency,
V = the volume or unit weight,
T_s = the sample count duration, and
Y = the fractional chemical recovery obtained for the tracer or carrier.

Other terms, as may be required (e.g., gamma abundance), can be used in the denominator.

3.13 *Supervisor* — A Laboratory (UC) employee who is a group, task, or project leader or manager.

3.14 *Total propagated uncertainty (TPU)* — The TPU associated with reported results shall be one sigma, 68% confidence interval. The TPU shall include reasonable and appropriate systematic uncertainties associated with analysis in addition to the uncertainty from contained statistics.

4.0 BACKGROUND AND PRECAUTIONS

n/a

5.0 EQUIPMENT

n/a

6.0 PROCEDURE

Note: Deviations from SOPs are made in accordance with QP-4.2.

6.1 General Procedure for Data Validation

6.1.1 The validator or the validator's deputy will pick up client data package at the Field Support Facility (FSF). The client data package is released to the validator or the validator's deputy under chain-of-custody. The validator or the validator's deputy is responsible for the

client data package until it is returned to the FSF under chain-of-custody.

6.1.2 The **validator or the validator's deputy** will log the client data package into the tracking database.

6.1.3 When the client data package is ready for validation, the **validator** will photocopy the Sample-Result Summary Forms (usually labeled Form 1) and the chain-of-custody documentation from the client's hard copy report. These forms will accompany the Baseline Validation Checklist (Attachment A).

6.1.4 The **validator** will include the following portions of the Baseline Validation Checklist (Attachment A) in the validation package:

- a validation cover sheet;
- the validation qualifiers that pertain to analyses validation;
- reason codes for validation qualifiers that pertain to analyses validation;
- the pertinent analyses checklist;
- under "Other Items Needed for the Completeness Check", the pertinent analyses section; and
- Table 6.2-1, if a volatile organic analysis, and/or Table 6.2-2, if a semivolatile organic analysis.

6.1.5 The **validator** completes the following information on the Validation Cover Sheet:

- the sample delivery group/request number (SDG/RN),
- the name of the contract laboratory,
- the validator's name,
- the name of the company for whom the validator works,
- the validation date,
- the applicable analytical suite(s), and
- the validator's dated signature.

6.1.6 The **validator** will also initial and date the analysis checklist pages and the photocopied pages of the Sample-Result Summary Form (Form 1).

6.1.7 The **validator** uses the forms photocopied from the client data package as the basis for the baseline analytical data validation with no examination of additional hard copy data—except where specifically indicated in the following baseline analytical data validation procedure. The validator places each set of qualifiers and reason codes on the right side of the Sample Result Summary Form in such a manner that

it is apparent that the qualifier and reason code are a set and that they apply, as a set, to either a specific analyte or group of analytes.

6.1.8 If the proper forms are not present, the **validator** contacts the contract laboratory and requests the missing forms.

6.1.8.1 The contract laboratory has three working days to FAX or send the missing forms to the validator unless the validator negotiates another turnaround time.

6.1.8.2 If forms are unavailable, the **validator** attaches an "A" qualifier to all of the sample results and places a comment in the section of the Baseline Validation Checklist (Attachment A) that pertains to the missing form.

6.1.9 The validator returns the original data package and the completed Baseline Validation Checklist (Attachment A) to the Sample Management Office (SMO). The validator will keep one copy for his or her files, and give one copy to the qualifier/reason-code-entry personnel.

6.2 Volatile Data Validation

6.2.1 Instrument-Performance Check

Verify the presence of the instrument-performance check (BFB) and verify that the instrument-performance check was analyzed on the same date or within 12 hours of the sample analysis from information provided on the forms supplied by the contract laboratory. The Instrument-Performance Check Form for volatile analysis is usually labeled as CLP form V.

6.2.2 Calibration

6.2.2.1 Verify the presence of the initial- and continuing-calibration results. The Initial Calibration Results Form for volatile analysis is usually labeled as CLP form VI and the Continuing Calibration Results Form for volatile analysis is usually labeled as CLP form VII.

6.2.2.2 Verify that the instrument continuing calibration was analyzed on the same date or within 12 hours of the sample analysis from information provided on the forms supplied by the contract laboratory.

6.2.3 Internal Standards

The internal standard retention times and area counts to be reported by the contract laboratory are chlorobenzene, 1,4-difluorobenzene, and 1,4-dichlorobenzene.

6.2.3.1 Verify the presence of the internal standards for all the requested samples. The Internal Standards Results Forms for volatile analysis are supplied by the contract laboratory and are usually labeled as CLP form VIII.

6.2.3.2 Use the following logic diagram (Figure 6.2-1) to determine which, if any, Laboratory qualifiers and reason codes are attached to the sample results.

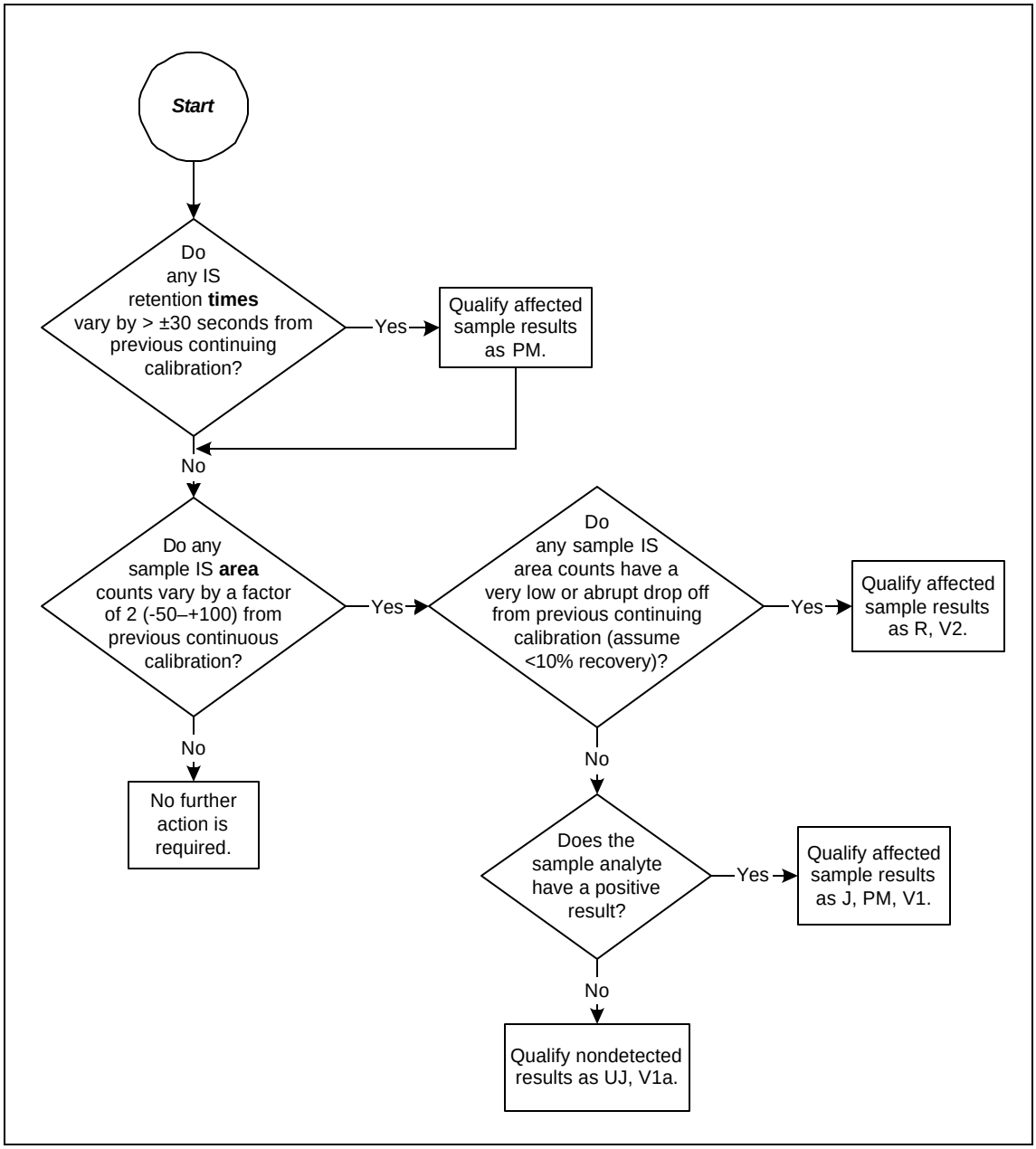

Figure 6.2-1. Logic diagram to determine applicable Laboratory qualifiers and reason codes for volatile analysis internal standard (IS) recovery results

6.2.4 System Monitoring Compounds

The system monitoring compounds (SMCs) recovery information is either summarized on the Sample Result Summary Forms or it may be located on a separate System Monitoring Compounds Recovery Results Form that contains all SMC information. The System Monitoring Compounds Recovery Results Form for volatile analysis is usually labeled as CLP form II.

6.2.4.1 Verify the presence of the SMC- (often referred to as "surrogates") recovery results from the information provided on the forms supplied by the contract laboratory. Table 6.2-1 lists the required surrogates for volatile analysis and the percent recovery criteria for both soil and waters.

Table 6.2-1
Surrogate Recovery Criteria for Volatile Analysis

SMC	Soil Recovery	Water Recovery
Toluene-d8	81%–117%	88%–110%
BFB	74%–121%	86%–115%
Dibromofluoromethane	80%–120%	86%–118%

6.2.4.2 Use the following logic diagram (Figure 6.2-2) to determine which, if any, Laboratory qualifiers and reason codes are attached to the sample results.

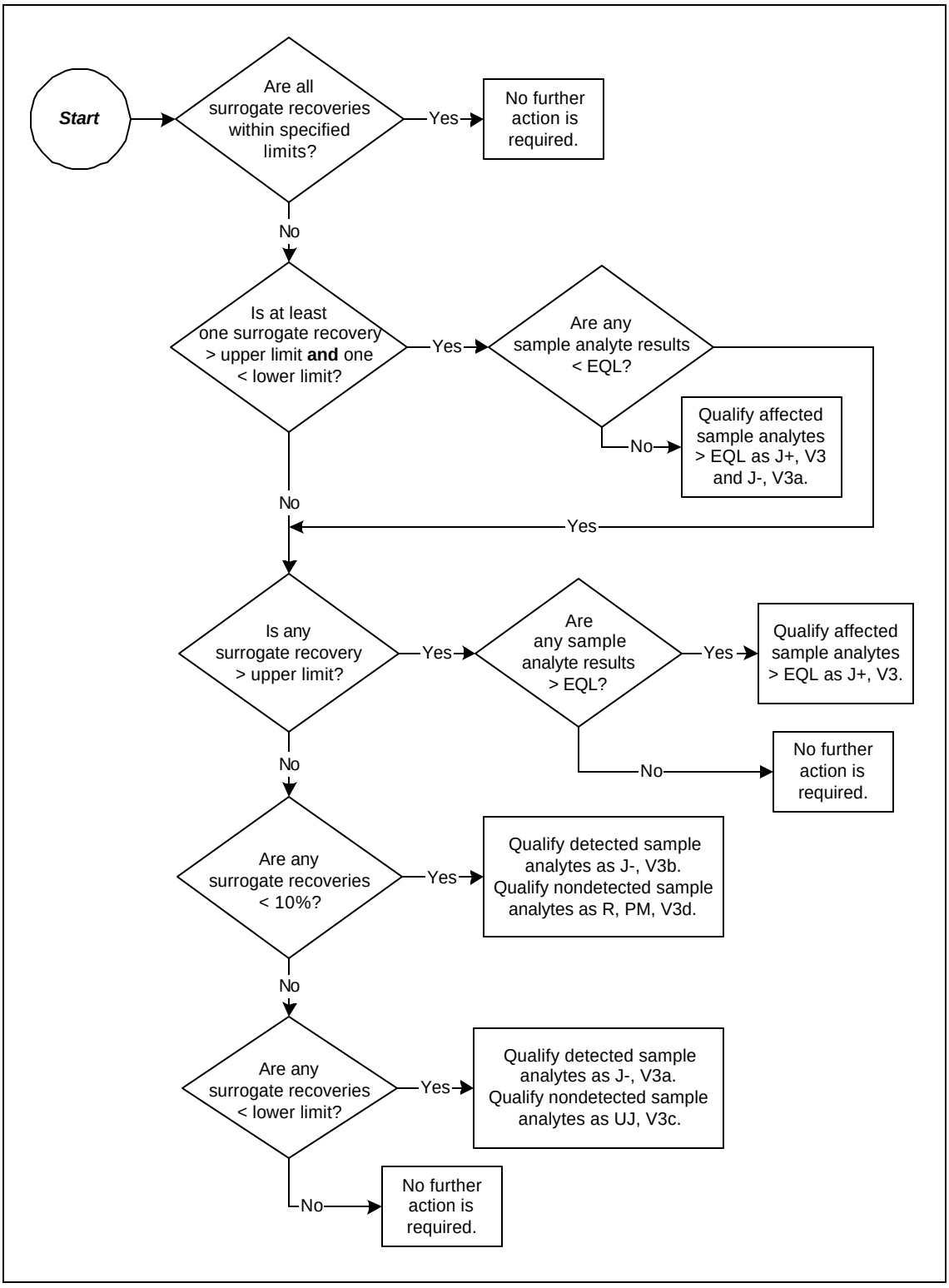

Figure 6.2-2. Logic diagram to determine applicable Laboratory qualifiers and reason codes for volatile analysis surrogate-recovery results

6.2.5 Method Blank

6.2.5.1 Verify that the method-blank results are present from the information provided on the Method Blank Results Form (usually labeled as CLP form IV for volatile analysis) supplied by the contract laboratory.

6.2.5.2 Manually compare the method-blank results to the contractually required EQLs listed in the statement of work (SOW).

6.2.5.3 Use the following logic diagram (Figure 6.2-3) to determine which, if any, Laboratory qualifiers and reason codes are attached to the sample results.

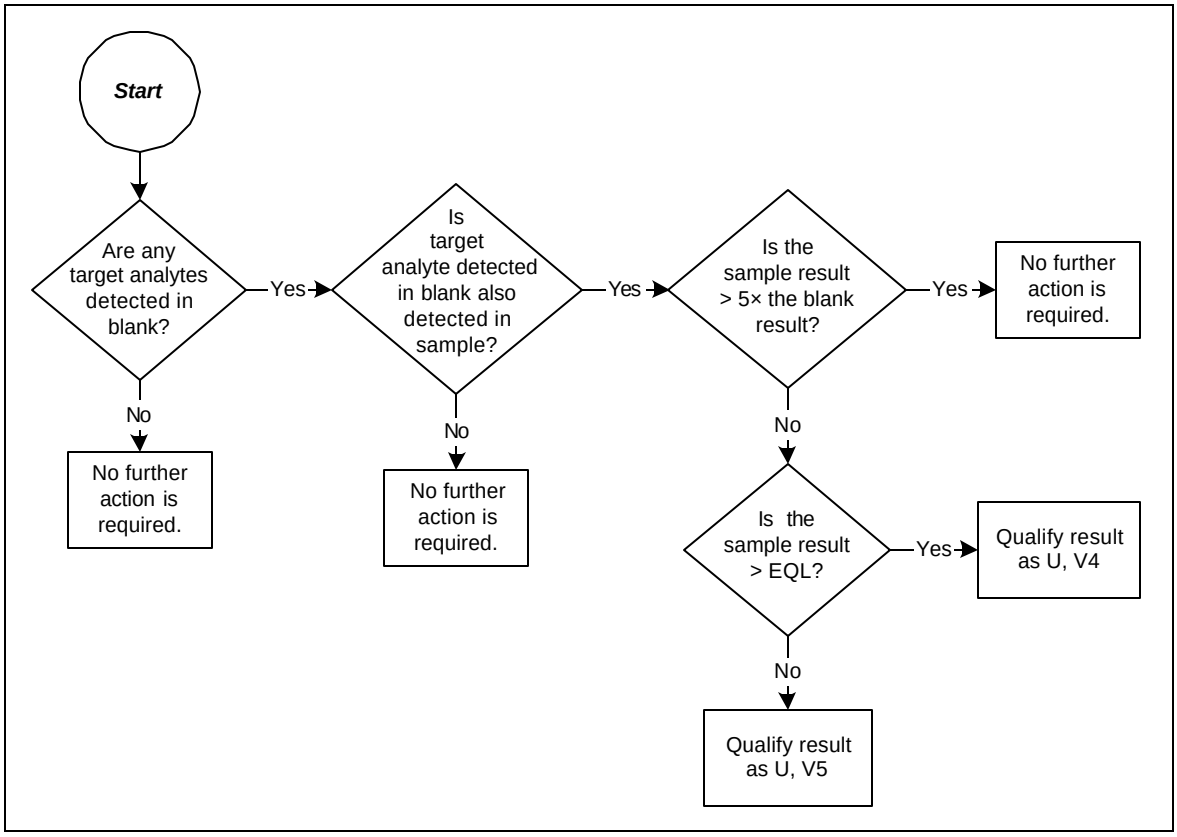

Figure 6.2-3. Logic diagram to determine applicable Laboratory qualifiers and reason codes for volatile analysis method blanks

6.2.6 Holding Time

The holding-time requirements for volatile analytes are analysis within 14 days of sample collection for soil samples and analysis within 7 days for water samples.

6.2.6.1 Calculate the holding time for a sample from the sampling date found on the chain-of-custody documentation that accompanies the data package. If the chain-of-custody documentation is missing from the data package, contact the Field Support Facility (FSF) and a copy will be immediately faxed to the validator.

6.2.6.2 If any holding times are not met for a sample qualify all analytes associated with that sample as PM. Place a comment at the bottom of the holding-time section of the Baseline Validation Checklist for Volatile Organic Analysis (Attachment A) that indicates each sample that was analyzed after its holding time had expired and by how many days the holding time was missed.

6.2.7 Tentatively Identified Compounds

6.2.7.1 If tentatively identified compounds (TICs) have not been requested, place an “N” following the Analysis Order Code on the chain-of-custody or agreement documentation.

6.2.7.2 If TICs are requested, then the validator verifies that the Tentatively Identified Compounds Report Form has been included in the data package. This report form generally follows the sample-results page.

6.2.7.3 If TICs are present in a sample, place a comment in the TIC’s section of the Baseline Validation Checklist for Volatile Organic Analysis (Attachment A).

6.2.8 Validation Completion

The **validator** is responsible for delivering the completed Baseline Validation Checklist(s) to the Sample Management Office (SMO) with the data package after the completion of the validation process.

6.3 Semivolatile Data Validation

6.3.1 Instrument-Performance Check

Verify the presence of the instrument-performance check (DFTPP) and verify that the instrument-performance check was analyzed on the same date or within 12 hours of the sample analysis from the information provided on the forms supplied by the contract laboratory. The Instrument-Performance Check Form for semivolatile analysis is usually labeled as CLP form V.

6.3.2 Calibration

6.3.2.1 Verify the presence of the initial- and continuing-calibration results. The Initial Calibration Results Form is usually labeled as CLP form VI and the Continuing Calibration Results Form is usually labeled as CLP form VII.

6.3.2.2 Verify that the instrument continuing calibration was analyzed on the same date or within 12 hours of the sample analysis from the information provided on the forms supplied by the contract laboratory.

6.3.3 Internal Standards

The internal standard area counts and retention times to be reported by the contract laboratory are 4-dichlorobenzene, naphthalene, acenaphthene, phenathrene, chrysene, and perylene.

6.3.3.1 Verify the presence of the internal standards for all the requested samples. The Internal Standards Results Form supplied by the contract laboratory is usually labeled as CLP form VIII.

6.3.3.2 Use the following logic diagram (Figure 6.3-1) to determine which, if any, Laboratory qualifiers and reason codes are attached to the sample results.

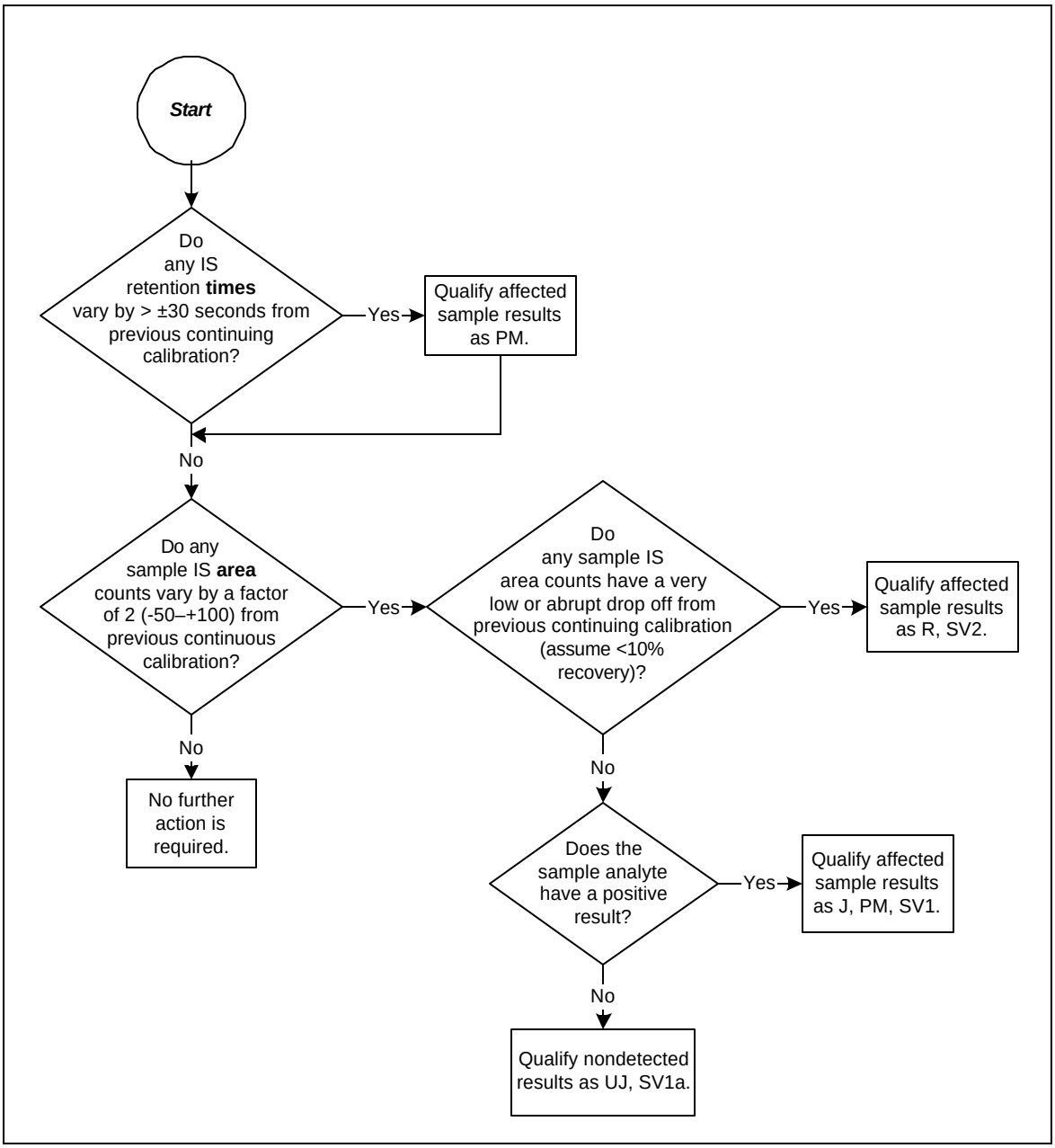

Figure 6.3-1. Logic diagram to determine applicable Laboratory qualifiers and reason codes for semivolatile organic analysis internal standard (IS) recovery results

6.3.4 System Monitoring Compounds

The system monitoring compounds (SMCs) recovery information is either summarized on the results form or it may be located on a separate System Monitoring Compounds Recovery Results Form (usually labeled as CLP form II for semivolatile analysis) that contains all SMC information.

6.3.4.1 Verify the presence of the SMC- (often referred to as “surrogates”) recovery results from the information provided on the forms supplied by the contract laboratory. Table 6.3-1 lists the required surrogates for semivolatile analysis and the percent recovery criteria for both soil and waters.

Table 6.3-1
Surrogate Recovery Criteria for Semivolatile Analysis

Surrogate	Soil Recovery	Water Recovery
nitrobenzene	23%–120%	35%–114%
2-fluorobiphenyl	30%–115%	43%–116%
p-terphenyl	18%–137%	33%–141%
phenol	24%–113%	10%–94%
2-fluorophenol	25%–121%	21%–100%
2,4,6-tribromophenol	19%–122%	10%–123%

6.3.5 Use the following logic diagram (Figure 6.3-2) to determine which, if any, Laboratory qualifiers and reason codes are attached to the sample results.

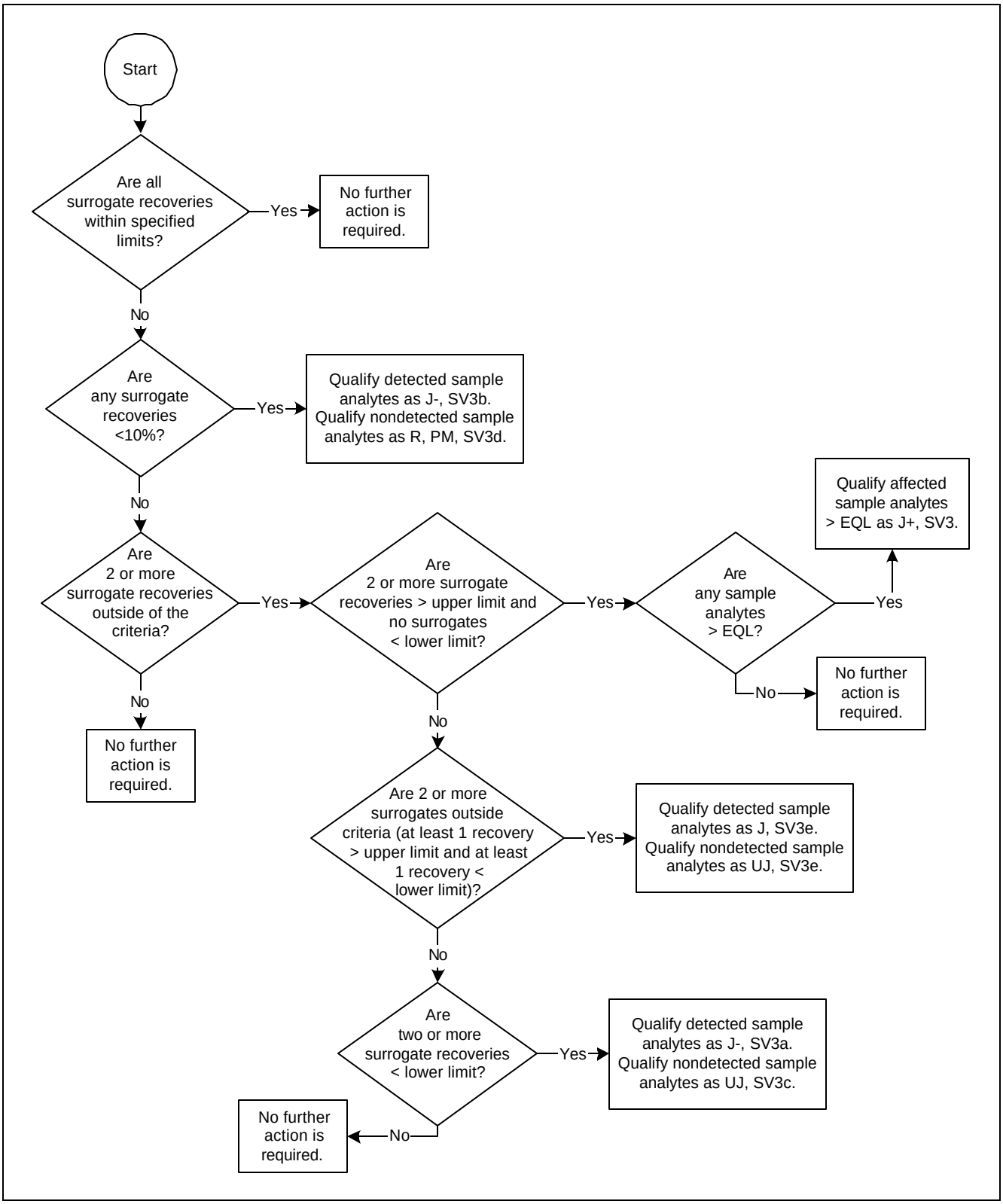

Figure 6.3-2. Logic diagram to determine applicable Laboratory qualifiers and reason codes for semivolatile analysis surrogate-recovery results

6.3.6 Method Blank

6.3.6.1 Verify that the method blank results are present from the information provided on the Method Blank Results Form (usually labeled as CLP form IV for semivolatile analysis) supplied by the contract laboratory.

6.3.6.2 Manually compare the method-blank results to the contractually required EQLs listed in the statement of work (SOW).

6.3.6.3 Use the following logic diagram (Figure 6.3-3) to determine which, if any, Laboratory qualifiers and reason codes are attached to the sample results.

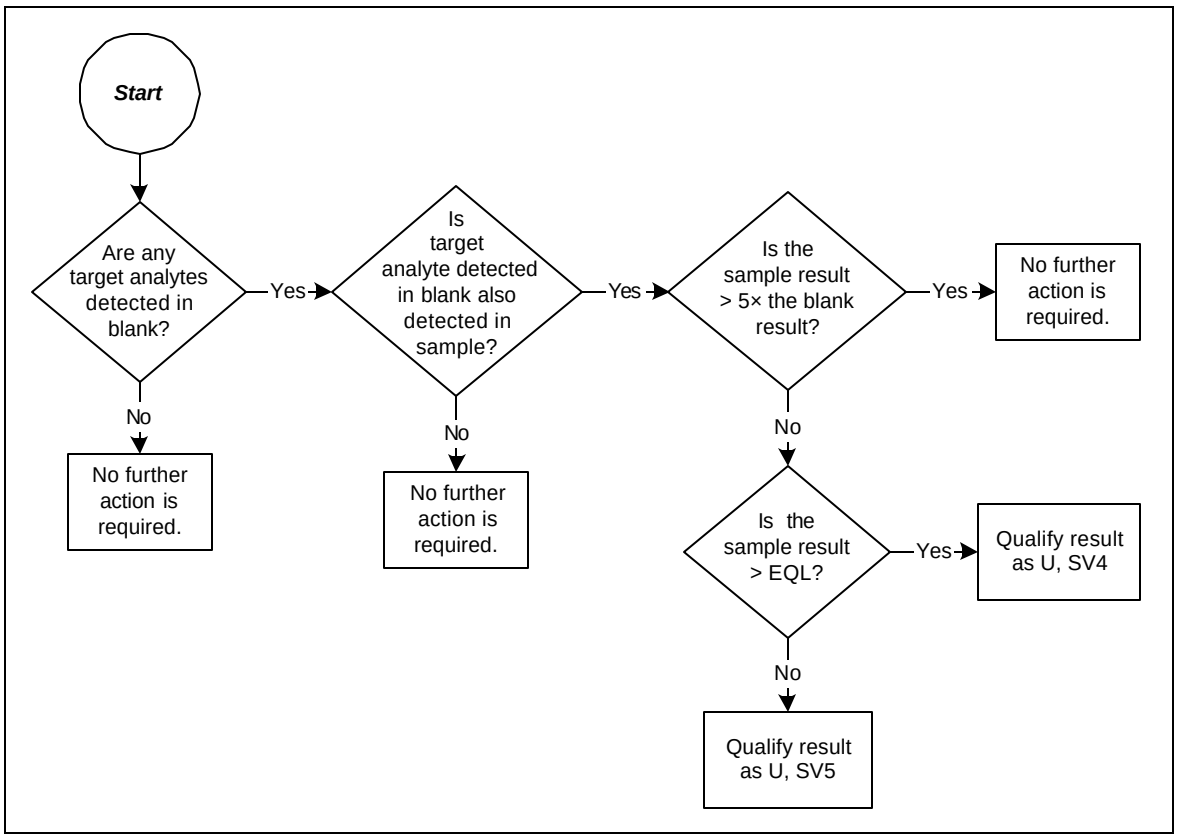

Figure 6.3-3. Logic diagram to determine applicable Laboratory qualifiers and reason codes for semivolatile analysis method-blank results

6.3.7 Holding Time

The holding time requirements for semivolatile analytes are extraction within 14 days of sample collection for soil samples and 7 days for water samples and analyses within 40 days of extraction.

6.3.7.1 Calculate the holding time for a sample from the sampling date found on the chain-of-custody documentation that

accompanies the data package. If the chain-of-custody documentation is missing from the data package, contact the Field Support Facility (FSF) and a copy will be immediately faxed to the validator.

6.3.7.2 If any holding times are not met for a sample qualify all analytes associated with that sample as PM. Place a comment at the bottom of the holding-time section of the Baseline Validation Checklist for Semivolatile Organic Analysis (Attachment A) that indicates each sample that was analyzed after its holding time had expired and by how many days the holding time was missed.

6.3.8 Tentatively Identified Compounds

6.3.8.1 If tentatively identified compounds (TICs) have not been requested, place an “N” following the Analysis Order Code on the chain-of-custody or agreement documentation.

6.3.8.2 If TICs are requested, then the validator verifies that the Tentatively Identified Compounds Report Form has been included in the data package. This report form generally follows the sample-results page.

6.3.8.3 If TICs are present in a sample, place a comment in the TIC’s section of the Baseline Validation Checklist for Semivolatile Organic Analysis (Attachment A).

6.3.9 Validation Completion

The **validator** is responsible for delivering the completed Baseline Validation Checklist(s) to the Sample Management Office (SMO) with the data package after the completion of the validation process.

6.4 Organochlorine/Pesticide/Aroclor Data Validation

6.4.1 Calibration

Verify the presence of the initial- and daily-calibration results from the information provided on the forms supplied by the contract laboratory. Generally, the Initial Calibration Results Form for organochlorine/pesticide/aroclor analysis is labeled as CLP form VI and the Daily Calibration Results Form for organochlorine/pesticide/aroclor analysis is labeled as CLP form VII.

6.4.2 System Monitoring Compounds

The system monitoring compounds (SMCs) recovery information is either summarized on the results form or it may be located on a separate System Monitoring Compounds Recovery Results Form

that contains all surrogate information (generally labeled as CLP form II for organochlorine/pesticide/aroclor analysis).

6.4.2.1 Verify the presence of the SMC- (often referred to as “surrogates”) recovery results from the information provided on the information supplied by the contract laboratory. The surrogates, tetrachloro-m-xylene and decachlorbiphenyl are required. The recovery criteria for both surrogates is 50%–160%.

6.4.2.2 Use the following logic diagram (Figure 6.4-1) to determine which, if any, Laboratory qualifiers and reason codes are attached to the sample results.

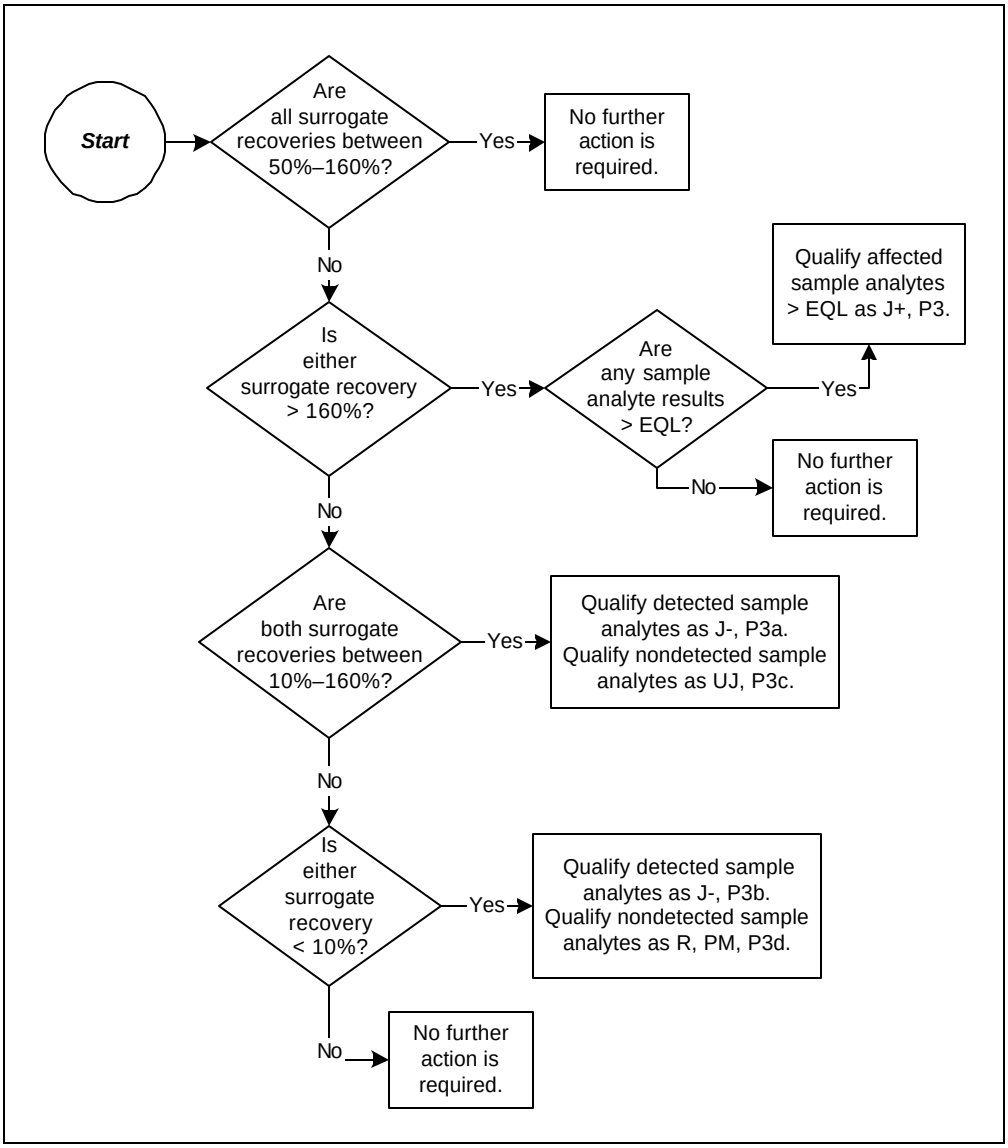

Figure 6.4-1. Logic diagram to determine applicable Laboratory qualifiers and reason codes for organochlorine/pesticide/aroclor analysis surrogate-recovery results

6.4.3 Retention-Time Windows

Compare the retention time for the sample surrogates to the retention time from the daily calibration of the surrogates that preceded the sample analysis.

6.4.3.1 The contract laboratories do not generally provide a summary form for retention-time windows. The retention-time information is generally found in the raw data. To further complicate matters, the raw data does not typically contain Laboratory sample ID numbers. Therefore, the validator must obtain the contract laboratory sample numbers from the Sample Result Summary Forms and use those numbers to find the retention-time information in the raw data.

6.4.3.2 Find the daily-calibration ID associated with the samples, which precedes either the contract-laboratory ID or Laboratory ID as reported on the run log. By using the run logs, the validator can then locate the ID in the raw data.

6.4.3.3 Use the surrogate's retention time from the appropriate daily calibration to calculate the retention-time windows. The retention time window should not shift by more than ± 0.05 minutes. If either of the sample's surrogate retention times shift by greater than ± 0.05 minutes, the validator qualifies all of the sample results as PM.

6.4.4 Method Blank

6.4.4.1 Verify the presence of the method blank results from the information provided on the Method Blank Results Form (usually labeled as CLP form IV for organochlorine/ pesticide/aroclor analysis) supplied by the contract laboratory.

6.4.4.2 Manually compare the method-blank results to the contractually required EQLs listed in the statement of work (SOW).

6.4.4.3 Use the following logic diagram (Figure 6.4-2) to determine which, if any, Laboratory qualifiers and reason codes are attached to the sample results.

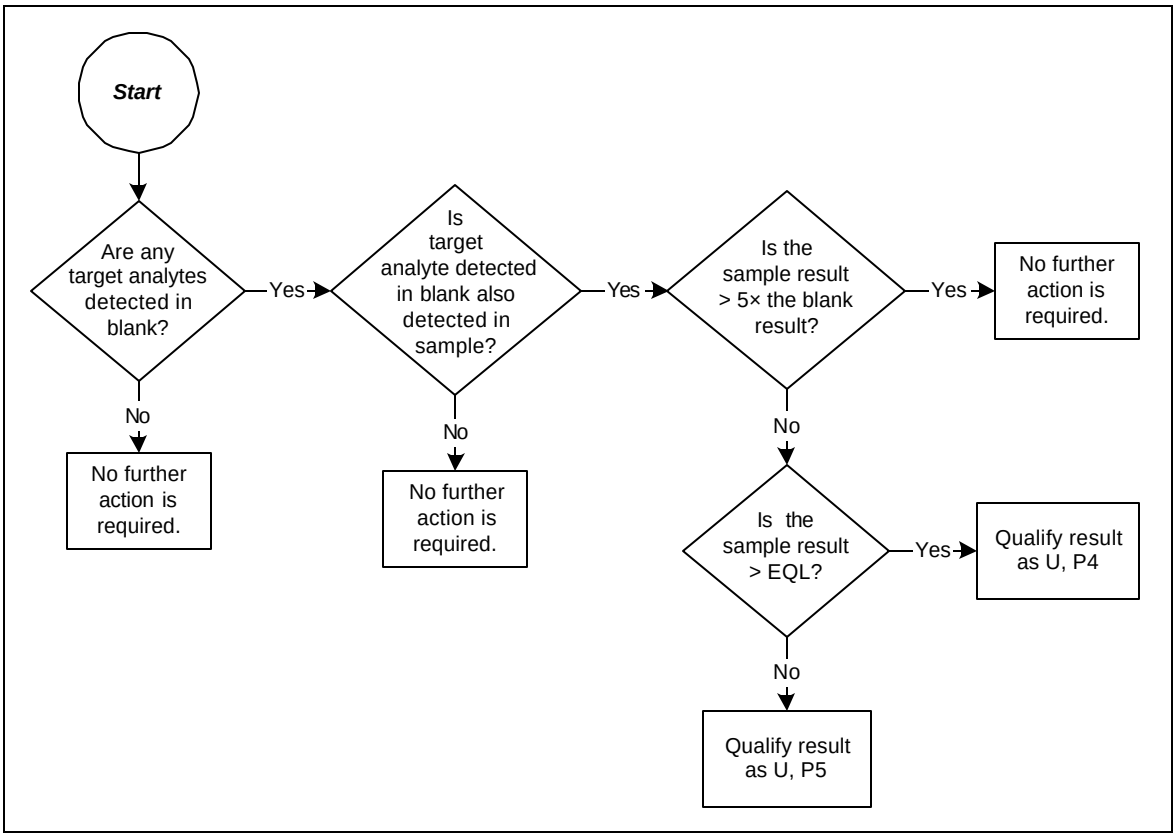

Figure 6.4-2. Logic diagram to determine applicable Laboratory qualifiers and reason codes for organochlorine/pesticide/aroclor analysis method blank results

6.4.5 Breakdown Criteria

6.4.5.1 If only aroclors (polychlorinated biphenyls—PCBs) are requested, then breakdown criteria are not relevant. The validator, in this case only, puts “NA” (for “not analyzed”) in the “Qualifiers Applied” column of this section of the Baseline Validation Checklist for Organochlorine/Pesticide/Aroclor Analysis (Attachment A). No further action is required.

6.4.5.2 Where breakdown criteria are required, the validator verifies that the contract laboratory reported the percent recoveries. The recoveries for the breakdown criteria can be found either summarized on a Breakdown Criteria Recoveries Form or, as is often the case, in the raw data. The break-down criteria are = 20% recovery for either 4,4’-DDT or endrin or = 30% recovery for a combined breakdown.

6.4.6 Holding Time

The holding time requirement for organochlorine/pesticide/aroclor analytes are extraction within 14 days of sample collection for soil

and 7 days for water samples and analyses within 40 days of extraction.

6.4.6.1 Calculate the holding time for a sample from the sampling date found on the chain-of-custody documentation that accompanies the data package. If the chain-of-custody documentation is missing from the data package, contact the Field Support Facility (FSF) and a copy will be immediately faxed to the validator.

6.4.6.2 If any holding times are not met for a sample qualify all analytes associated with that sample as PM. Place a comment at the bottom of the holding-time section of the Baseline Validation Checklist for Organochlorine/Pesticide/ Aroclor Analysis (Attachment A) that indicates each sample that was analyzed after its holding time had expired and by how many days the holding time was missed.

6.4.7 Validation Completion

The **validator** is responsible for delivering the completed Baseline Validation Checklist(s) to the Sample Management Office (SMO) with the data package after the completion of the validation process.

6.5 High Explosive Data Validation

6.5.1 Calibration

Verify the presence of the initial- and daily-calibration results from the information provided on the forms supplied by the contract laboratory.

6.5.2 Laboratory Control Sample

6.5.2.1 Verify the presence of the laboratory control sample (LCS) sample recoveries from the information provided on the forms supplied by the contract laboratory. A blank-spike result is often reported instead of a LCS. This is equivalent nomenclature and no qualification needs to be applied. Provide a comment at the end of the LCS section of the Baseline Validation Checklist for High Explosives Analysis (Attachment A) that the LCS was called a blank spike.

6.5.2.2 The statement of work (SOW) does not require that the LCS (or blank spike) contain all analytes of interest. A known amount of 7–10 analytes should be present in the LCS (or blank spike). When the LCS (or blank spike) does not contain all the analytes of interest, only those analytes analyzed are qualified. Provide a comment at the end of the LCS section of the Baseline Validation Checklist for High Explosives Analysis

(Attachment A) that lists the LCS (blank spike) analytes present.

6.5.2.3 If less than 7 analytes are present, apply an “A” qualifier to all the sample results and attach a comment to the bottom of the LCS (or blank spike) section of the Baseline Validation Checklist for High Explosives Analysis (Attachment A) that explains that not enough analytes were spiked into the LCS (or blank spike) solution and lists the analytes present in the LCS (or blank spike).

6.5.2.4 Use the following logic diagram (Figure 6.5-1) to determine which, if any, Laboratory qualifiers and reason codes are attached to the sample results.

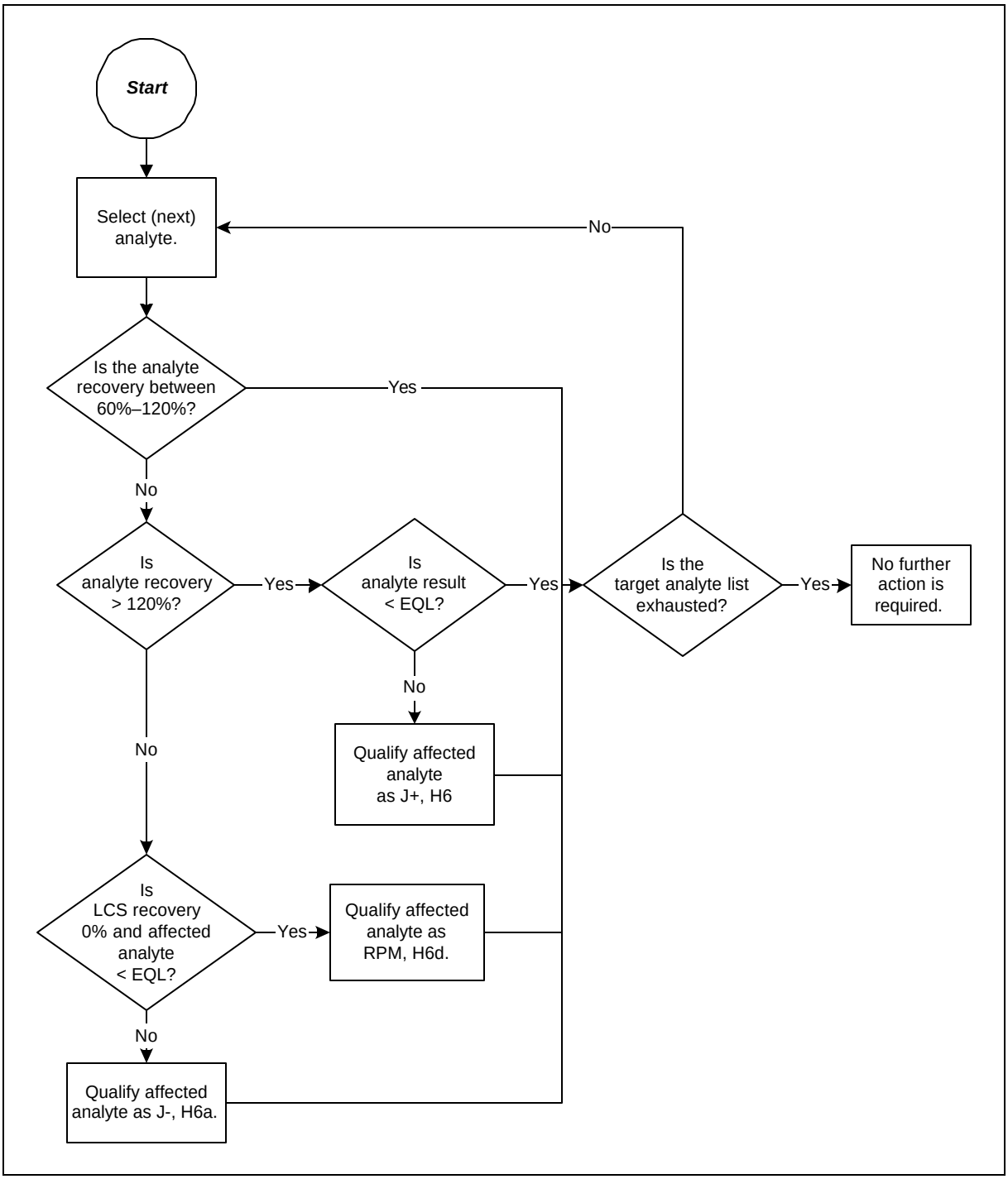

Figure 6.5-1. Logic diagram to determine applicable Laboratory qualifiers and reason codes for high explosives analysis LCS (or blank spike) recoveries

6.5.3 System Monitoring Compounds

The system monitoring compounds (SMCs) recovery information is either summarized on the results form or it may be located on a

separate System Monitoring Compounds Recovery Results Form that contains all surrogate information.

6.5.3.1 Verify the presence of the SMC- (often referred to as “surrogates”) recovery results from the information provided on the forms supplied by the contract laboratory.

6.5.3.2 The surrogate 3,4-dinitrotoluene is required and a second surrogate, 4-nitroaniline, is optional. At this time no recovery criterion is specified for either of these surrogates. It should be noted that high explosive surrogates were not required before September of 1995.

6.5.4 Method Blank

6.5.4.1 Verify that the method-blank results are present from the information provided on the Method Blank Results Form supplied by the contract laboratory.

6.5.4.2 Manually compare the method-blank results to the contractually required EQLs listed in the SOW.

6.5.4.3 Use the following logic diagram (Figure 6.5-2) to determine which, if any, Laboratory qualifiers and reason codes are attached to the sample results.

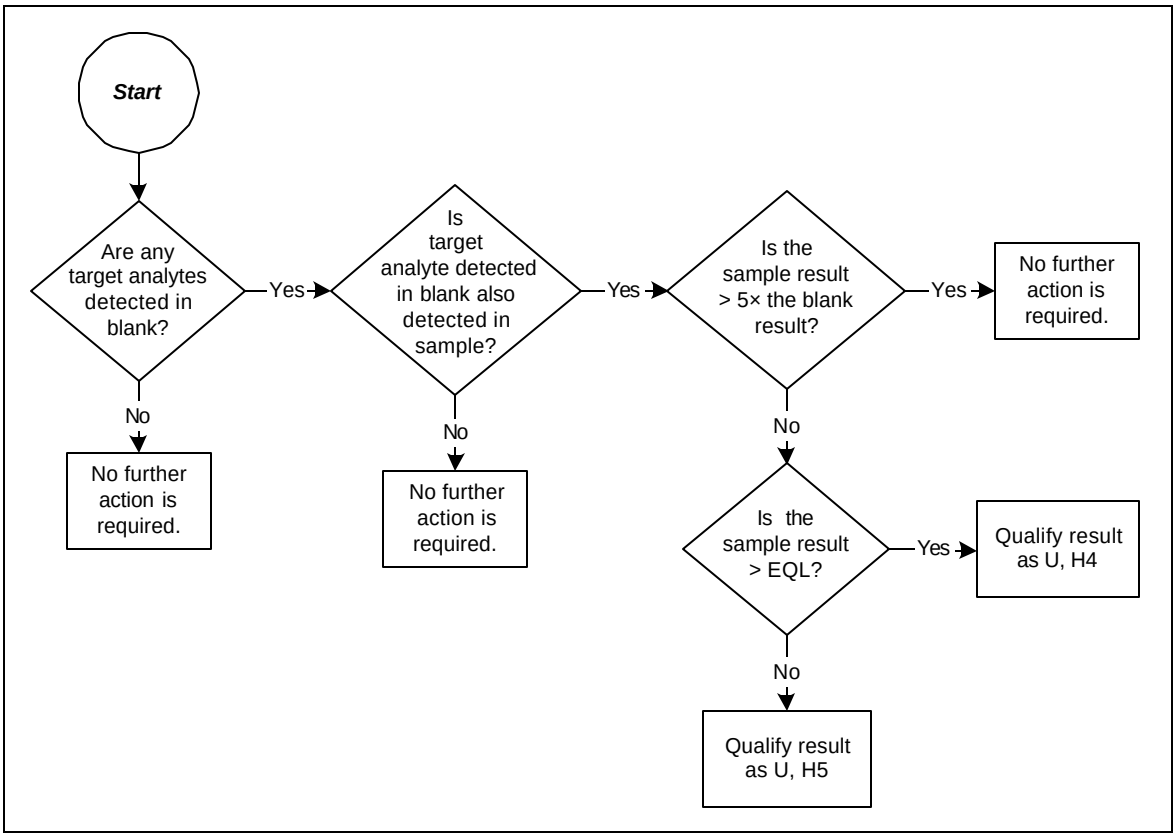

Figure 6.5-2. Logic diagram to determine applicable Laboratory qualifiers and reason codes for high explosives analysis method-blank results

6.5.5 Holding Time

The holding-time requirements for high explosives analytes are extraction within 14 days of sample collection for soil samples and 7 days for water samples and analyses within 40 days of extraction.

6.5.5.1 Calculate the holding time for a sample from the sampling date found on the chain-of-custody documentation that accompanies the data package. If the chain-of-custody documentation is missing from the data package, contact the Field Support Facility (FSF) and a copy will be immediately faxed to the validator.

6.5.5.2 If any holding times are not met for a sample qualify all analytes associated with that sample as PM. Place a comment at the bottom of the holding-time section of the Baseline Validation Checklist for High Explosives Analysis (Attachment A) that indicates each sample that was analyzed after its holding time had expired and by how many days the holding time was missed.

6.5.6 Validation Completion

The **validator** is responsible for delivering the completed Baseline Validation Checklist(s) to the Sample Management Office (SMO) with the data package after the completion of the validation process.

6.6 Inorganic Data Validation

6.6.1 Calibration

Verify the presence of the initial- and daily-calibration results from the information provided on the forms supplied by the contract laboratory. Generally, for inorganic analysis the Initial Calibration Results Form is labeled as CLP form II and the Daily Calibration Results Form is labeled as CLP form II.

6.6.2 Blanks

6.6.2.1 Verify the presence of the initial- and continuing-calibration blanks and preparatory blanks' results from the information provided on the forms supplied by the contract laboratory Generally, the Preparatory Blank Results Form for inorganic analysis is labeled as CLP form III.

6.6.2.2 Due to time constraints, only manually validate the preparation blank. Results greater than the instrument detection limits (IDL) found in the initial- and/or continuing-calibration blank are noted at the bottom of the blanks section of the Baseline Validation Checklist for Inorganic Analysis (Attachment A).

6.6.2.3 Manually compare the preparation-blank results to the contractually required EDLs listed in the statement of work (SOW).

6.6.2.4 Use the following logic diagram (Figure 6.6-1) to determine which, if any, Laboratory qualifiers and reason codes are attached to the sample results.

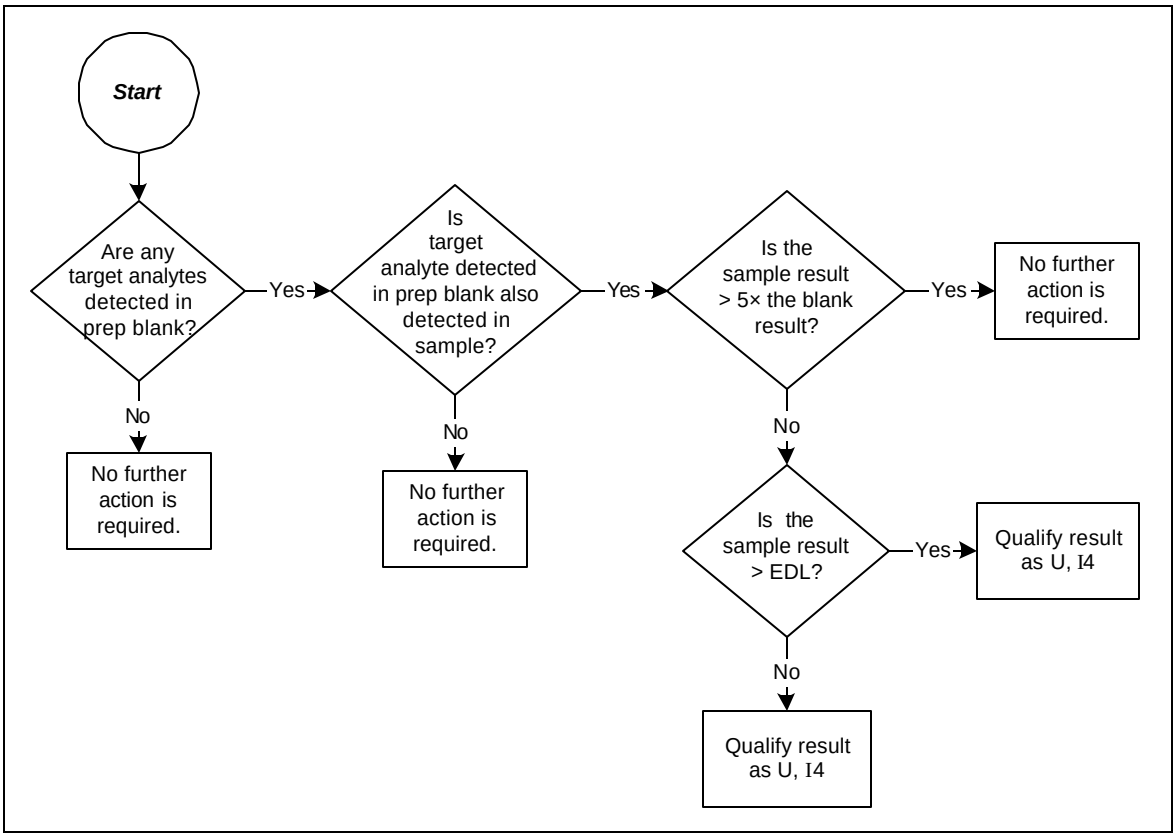

Figure 6.6-1. Logic diagram to determine applicable Laboratory qualifiers and reason codes for inorganic analysis preparation-blank results

6.6.3 ICP Interference Check Sample

6.6.3.1 Verify the presence of the ICP Interference Check Sample recoveries from the information provided on the forms supplied by the contract laboratory. For inorganic analysis, the ICP Interference Check Sample Recovery Results Form is generally labeled as CLP form IV.

6.6.3.2 If sample was analyzed for lead by graphite-furnace atomic absorption, no interference-check result is required. Note this information at the end of the ICP Interference Check Sample Recoveries section of the Baseline Validation Checklist for Inorganic Analysis (Attachment A).

6.6.3.3 Use the following logic diagram (Figure 6.6-2) to determine which, if any, Laboratory qualifiers and reason codes are attached to the sample results.

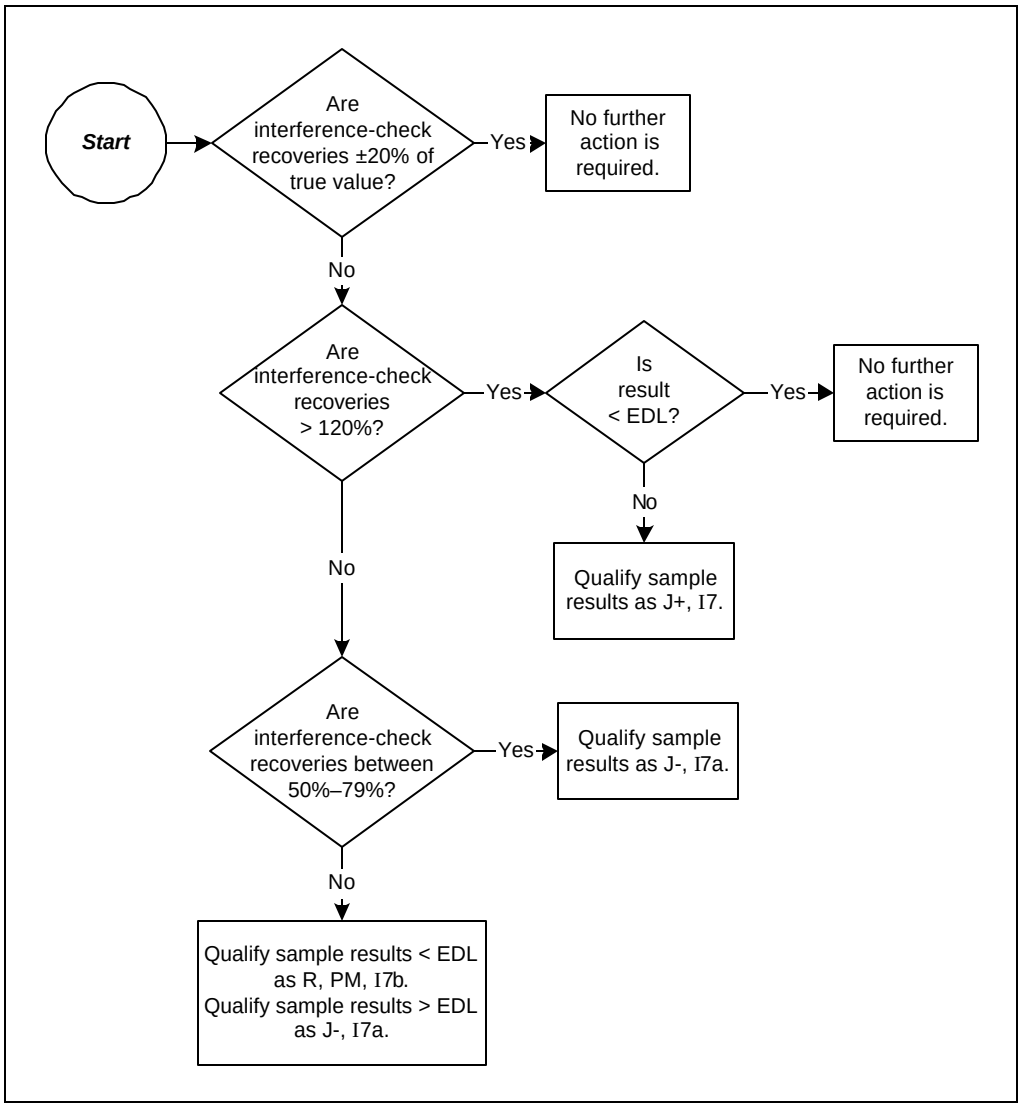

Figure 6.6-2. Logic diagram to determine applicable Laboratory qualifiers and reason codes for inorganic analysis interference-check spike sample results

6.6.4 Matrix Spike

6.6.4.1 Verify the presence of the matrix spike sample recoveries from the information provided on the forms supplied by the contract laboratory. For inorganic analysis, the Matrix Spike Recovery Results Form is generally labeled as CLP form V.

6.6.4.2 If a matrix spike was analyzed on a sample not associated with this request and no matrix spike was analyzed on a sample associated with this request, attach an “A” qualifier to all the samples and analyte results associated with this request. Include a comment at the bottom of the Matrix Spike Recovery Results section of the Baseline Validation Checklist

for Inorganic Analysis (Attachment A). No further qualification is required.

6.6.4.3 Use the following logic diagram (Figure 6.6-3) to determine which, if any, Laboratory qualifiers and reason codes are attached to the sample results.

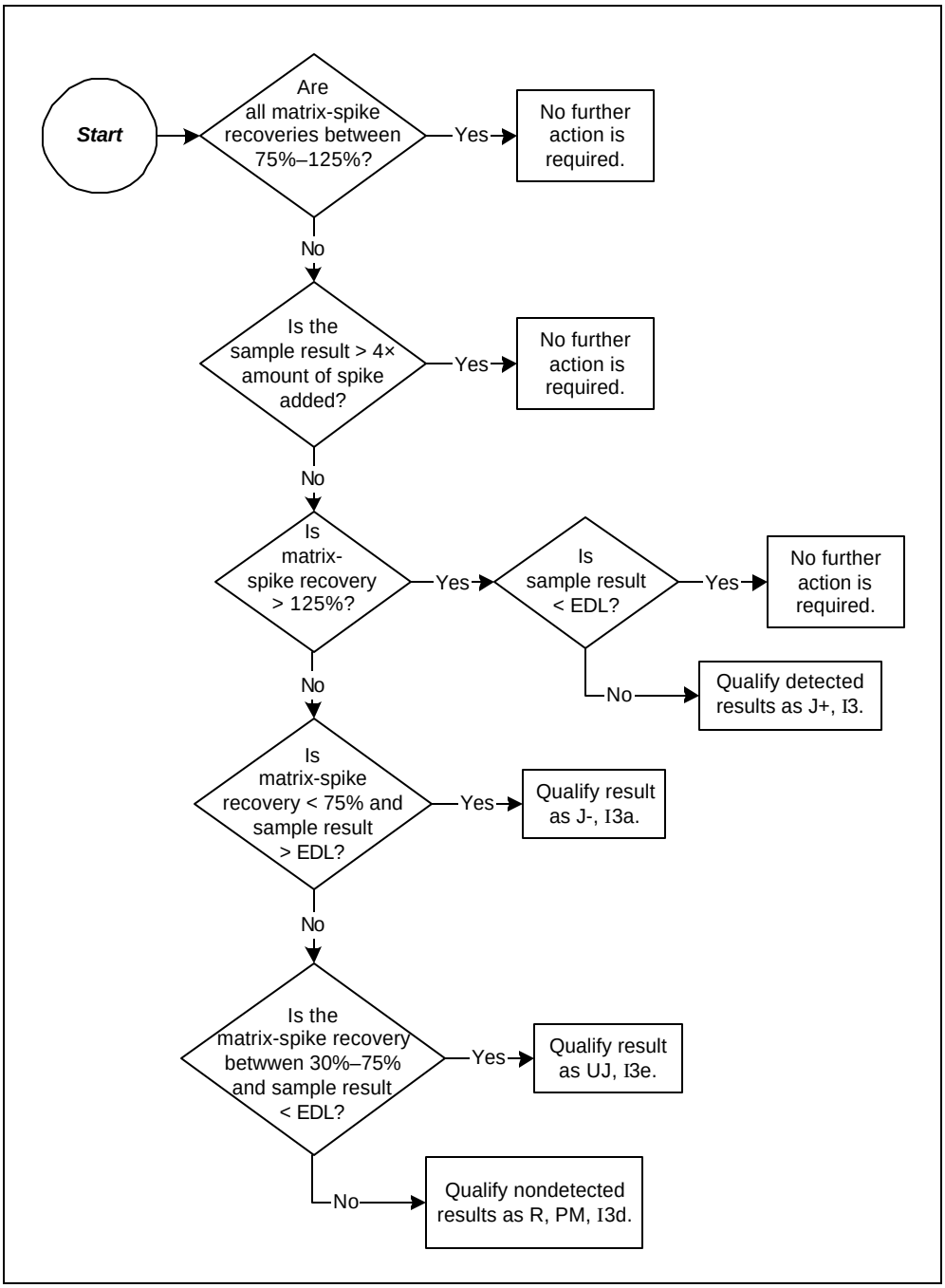

Figure 6.6-3. Logic diagram to determine applicable Laboratory qualifiers and reason codes for inorganic analysis matrix spike recovery results

6.6.5 Duplicates

6.6.5.1 Verify the presence of the duplicate-sample recoveries from the information provided on the forms supplied by the contract laboratory. For inorganic analysis, the Duplicate Sample Recovery Results Form is generally labeled as CLP form VI.

6.6.5.2 If a duplicate was analyzed on a sample not associated with this request and no duplicate was analyzed on a sample associated with this request, attach an “A” qualifier to all the samples and analyte results associated with this request. Include a comment at the bottom of the Duplicates section of the Baseline Validation Checklist for Inorganic Analysis (Attachment A). No further qualification is required.

6.6.5.3 Use the following logic diagram (Figure 6.6-4) to determine which, if any, Laboratory qualifiers and reason codes are attached to the sample results.

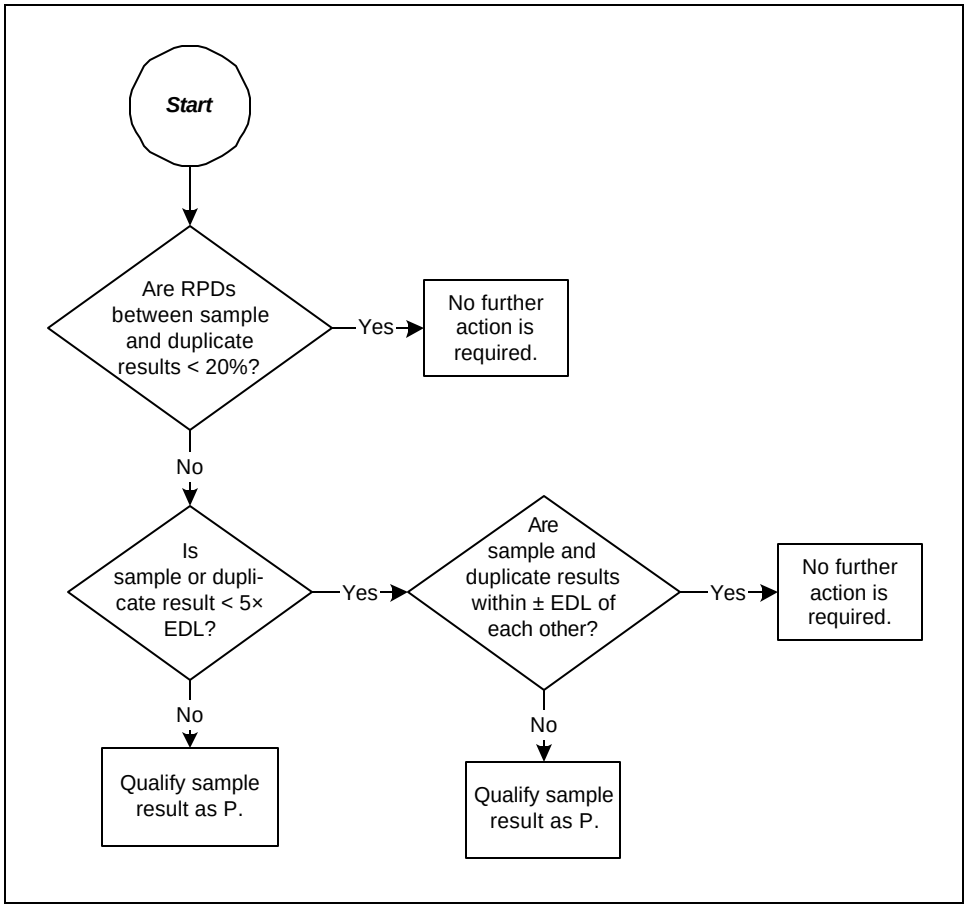

Figure 6.6-4. Logic diagram to determine applicable Laboratory qualifiers and reason codes for inorganic analysis duplicate results

6.6.6 Laboratory Control Sample

6.6.6.1 Verify the presence of the laboratory control sample (LCS) sample recoveries from the information provided on the forms supplied by the contract laboratory. For inorganic analysis, the Laboratory Control Sample Recovery Results Form is generally labeled as CLP form VII. For each aqueous sample request, an aqueous LCS must have been prepared and analyzed. For soil samples, if a solid LCS is available from a vendor, then its use is permitted. If a solid LCS is unavailable, an aqueous LCS must be used.

6.6.6.2 If solid samples were analyzed, place “see comment” in the “Qualifiers Applied” column of the LCS section of the Baseline Validation Checklist for Inorganic Analysis (Attachment A).

6.6.6.3 Compare the solid LCS results to the aqueous criteria and then note any analyses that do not meet the criteria at the bottom of the LCS section of the Baseline Validation Checklist for Inorganic Analysis (Attachment A).

6.6.6.4 Do not apply qualifications to solid LCS results that do not meet aqueous criteria as this information is provided only to aid in assessing possible data quality.

6.6.6.5 Use the following logic diagram (Figure 6.6-5) to determine which, if any, Laboratory qualifiers and reason codes are attached to the sample results. This diagram applies to aqueous results only.

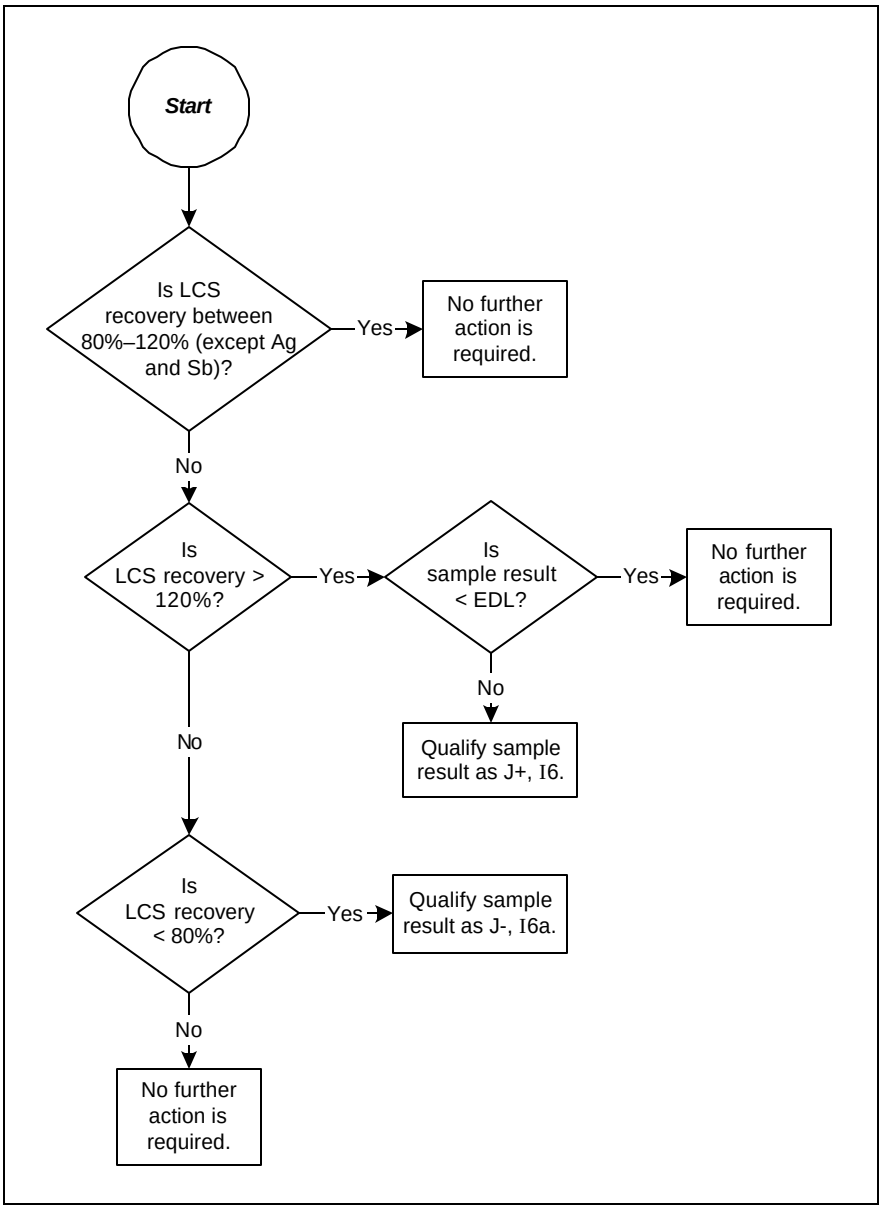

Figure 6.6-5. Logic diagram to determine applicable Laboratory qualifiers and reason codes for inorganic analysis LCS results

6.6.7 Holding Times

the validator must only check the holding times for Mercury and Cyanide aqueous sample results. The mercury holding time is 28 days. The cyanide holding time is 14 days.

6.6.7.1 Calculate the holding time for a sample from the sampling date found on the chain-of-custody documentation that accompanies the data package. If the chain-of-custody documentation is missing from the data package, contact the

Field Support Facility (FSF) and a copy will be immediately faxed to the validator.

6.6.7.2 If any holding times are not met for a sample, qualify all analytes associated with that sample as PM. Place a comment at the bottom of the holding-time section of the Baseline Validation Checklist for Inorganic Analysis (Attachment A) that indicates each sample that was analyzed after its holding time had expired and by how many days the holding time was missed.

6.6.8 Validation Completion

The **validator** is responsible for delivering the completed Baseline Validation Checklist(s) to the Sample Management Office (SMO) with the data package after the completion of the validation process.

6.7 Radiochemistry Data Validation

6.7.1 Laboratory Control Sample

6.7.1.1 Verify the presence of the laboratory control sample (LCS) sample recoveries from the information provided on the forms supplied by the contract laboratory. If the forms are not present, the validator must contact the contract laboratory and request the missing forms. If no forms are available, attach an “A” qualifier to all the sample results and place a comment in the LCS recovery results section of the Baseline Validation Checklist for Radiochemistry Analysis (Attachment A).

6.7.1.2 A blank spike result is often reported instead of LCS recovery results. This is equivalent nomenclature and no qualification is applied. Provide a comment at the end of the LCS recovery results section of the Baseline Validation Checklist for Radiochemistry Analysis (Attachment A) that the LCS was called a blank spike.

6.7.1.3 The statement of work (SOW) does not require that the LCS (or blank spike) contain all nuclides of interest. When the LCS (or blank spike) does not contain all the nuclides of interest, only those nuclides which were analyzed are qualified. Provide a comment at the end of the LCS recovery results section of the Baseline Validation Checklist for Radiochemistry Analysis (Attachment A) that lists the LCS (or blank spike) nuclides that were present.

6.7.1.4 If the tracer or carrier recovery is less than 10%, qualify the associated sample data as “P” for nondetected.

6.7.1.5 Use the following logic diagram (Figure 6.7-1) to determine which, if any, Laboratory qualifiers and reason codes are attached to the sample results.

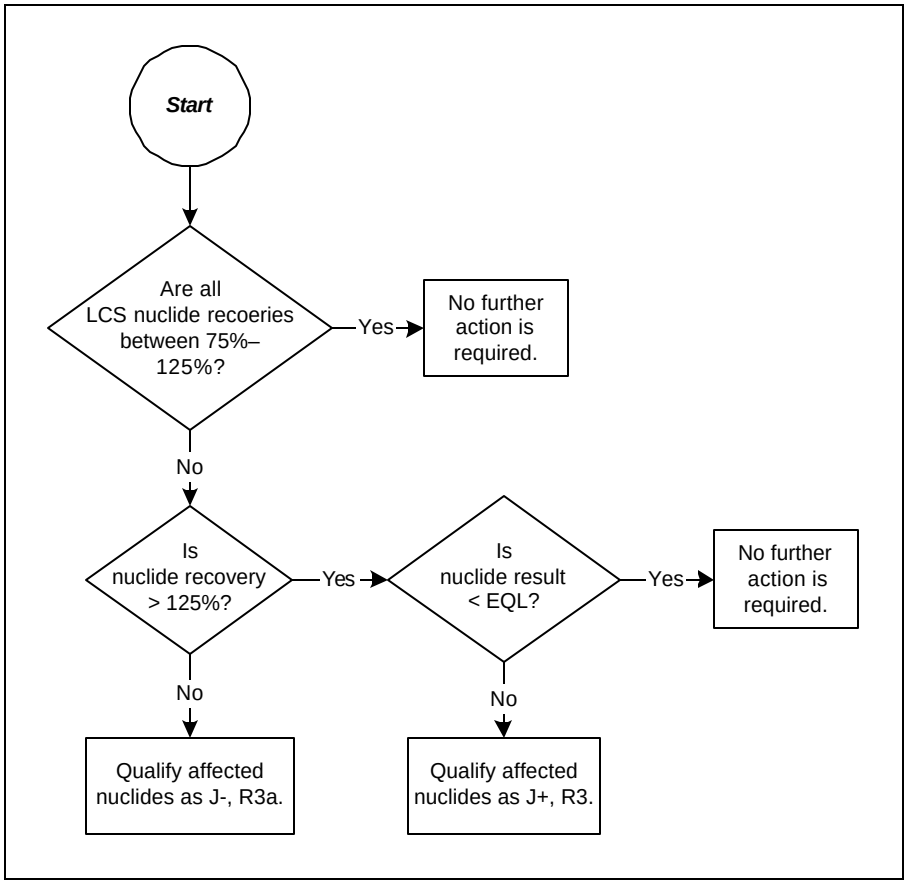

Figure 6.7-1. Logic diagram to determine applicable Laboratory qualifiers and reason codes for radiochemical analysis of LCS recovery results

6.7.2 Matrix Spike

6.7.2.1 The matrix-spike analysis is required on the following analyses:

- strontium-90 when a strontium-85 tracer or equivalent is not used;
- tritium by liquid scintillation;
- total uranium by kinetic phosphorescence analysis (KPA);
- all mass spectroscopy techniques;
- radium-226 analysis by other than tracer techniques; and

- all analyses of radium-228,thorium-234, and lead-210.

Note: Total uranium analysis by KPA is a nonroutine analysis method.

6.7.2.2 Validate the matrix-spike after the method-blank and EQL tests have been completed.

6.7.2.3 If a matrix spike was analyzed on a sample not associated with this request and no matrix spike was analyzed on a sample associated with this request, attach an “A” qualifier to all the samples and nuclide results associated with this request. Include a comment at the bottom of the matrix-spike section of the Baseline Validation Checklist for Radiochemistry Analysis (Attachment A). No further qualification is required.

6.7.2.4 In a situation where insufficient sample was provided to analyze both a duplicate and a matrix spike, the duplicate analysis shall take precedence. In the case narrative, the contract laboratory should include any reason why the matrix spike could not be analyzed. If no explanation was included in the case narrative, the validator must contact the contract laboratory and request that an amended case narrative be submitted via FAX.

6.7.2.5 Use the following logic diagram (Figure 6.7-2) to determine which, if any, Laboratory qualifiers and reason codes are attached to the sample results.

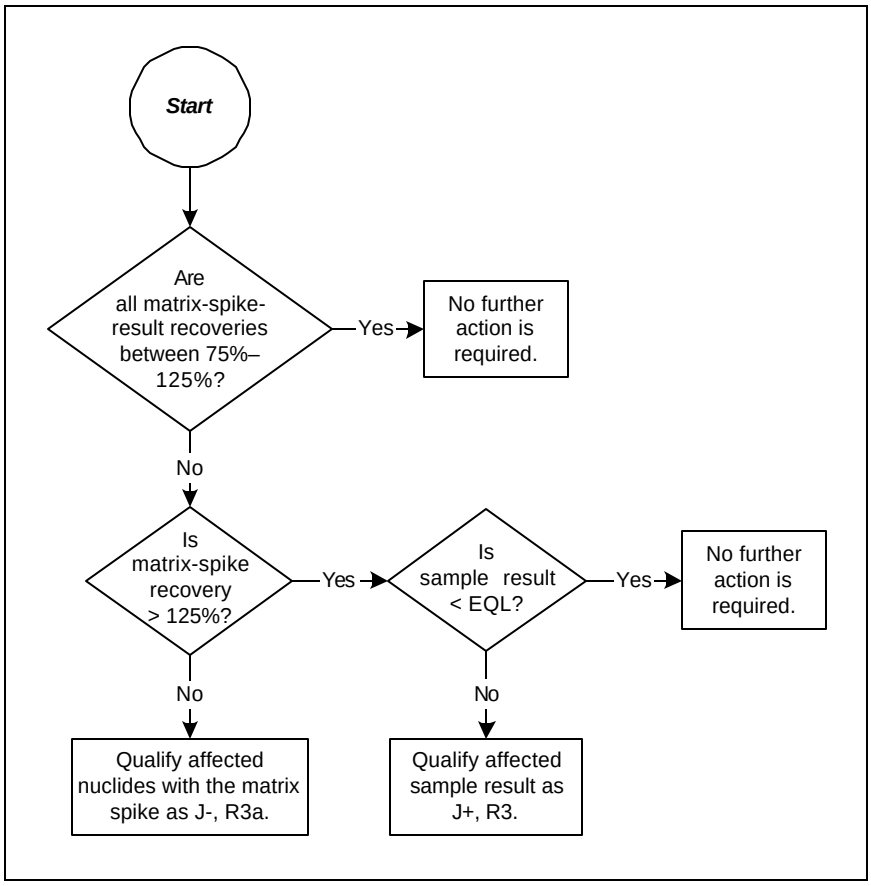

Figure 6.7-2. Logic diagram to determine applicable Laboratory qualifiers and reason codes for radiochemical analysis of matrix spike samples

6.7.3 Blanks

6.7.3.1 Verify the presence of the preparatory-blanks' results from the information provided on the forms supplied by the contract laboratory. Not all radiochemical analyses have required EQLs and, therefore, these analyses are treated differently than those with contract-specific EQLs.

6.7.3.2 Manually compare the method blank results to the contractually-required EQLs listed in the SOW.

6.7.3.3 Use the following logic diagram (Figure 6.7-3) to determine which, if any, Laboratory qualifiers and reason codes are attached to the sample results.

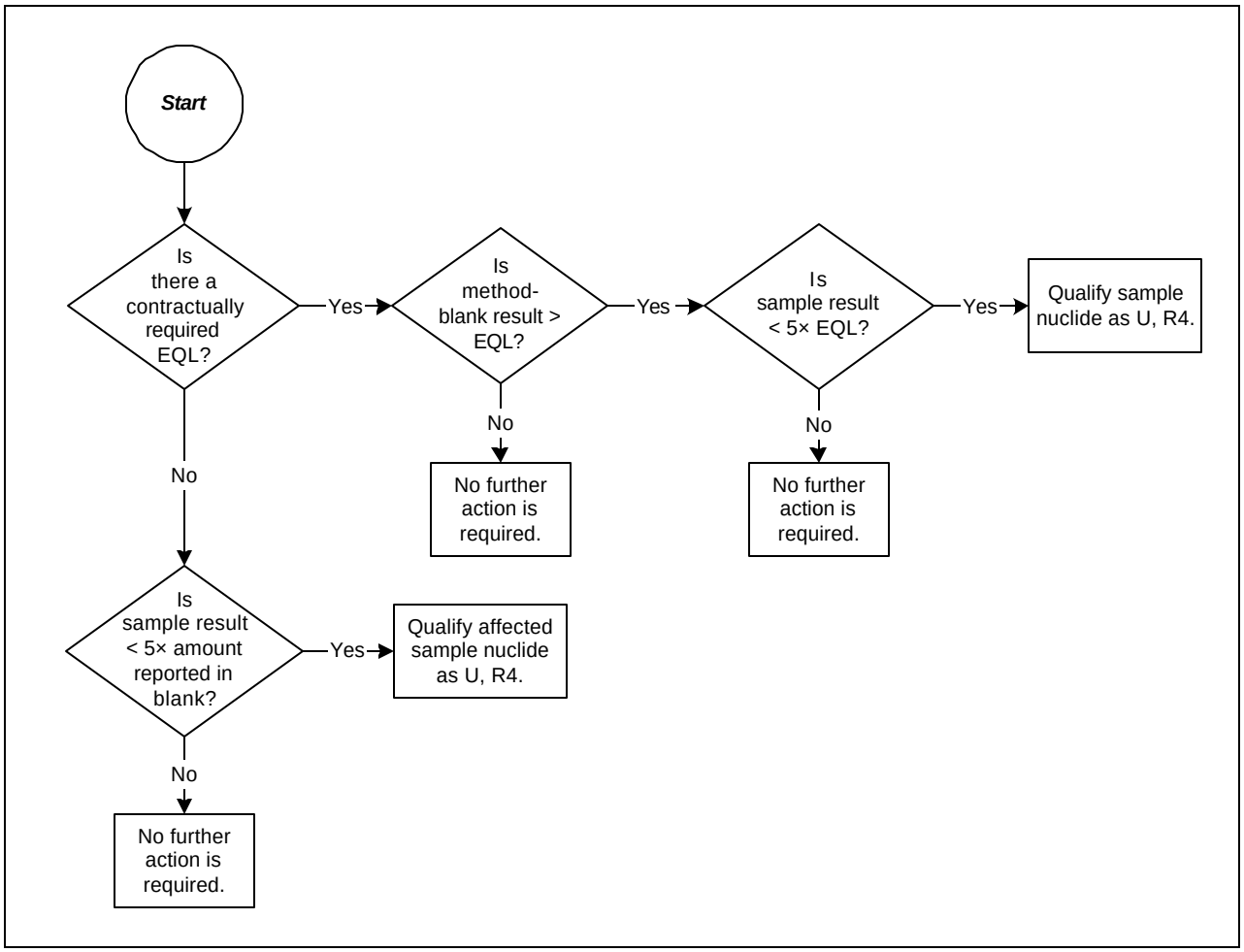

Figure 6.7-3. Logic diagram to determine applicable Laboratory qualifiers and reason codes for radiochemical analysis of method blank results

6.7.4 Duplicates

6.7.4.1 Verify the presence of the duplicate sample recoveries from the information provided on the forms supplied by the laboratory.

6.7.4.2 Validate the duplicate sample after the method-blank and the EQL tests have been completed.

6.7.4.3 If a duplicate was analyzed on a sample not associated with this request and no duplicate was analyzed on a sample associated with this request, attach an “A” qualifier to all the samples and nuclide results associated with this request. Include a comment at the bottom of the Duplicates section of the Baseline Validation Checklist for Radiochemistry Analysis (Attachment A). No further qualification is required.

6.7.4.4 In a situation where insufficient sample was provided to analyze both a duplicate and a matrix spike, the duplicate analysis shall take precedence. If insufficient sample was provided to analyze either the duplicate or the matrix spike, in the case narrative, the contract laboratory should include any reason why the duplicate could not be analyzed. If no explanation was included in the case narrative, the validator must contact the contract laboratory and request that an amended case narrative be submitted via FAX.

6.7.4.5 Use the following logic diagram (Figure 6.7-4) to determine which, if any, Laboratory qualifiers and reason codes are attached to the sample results.

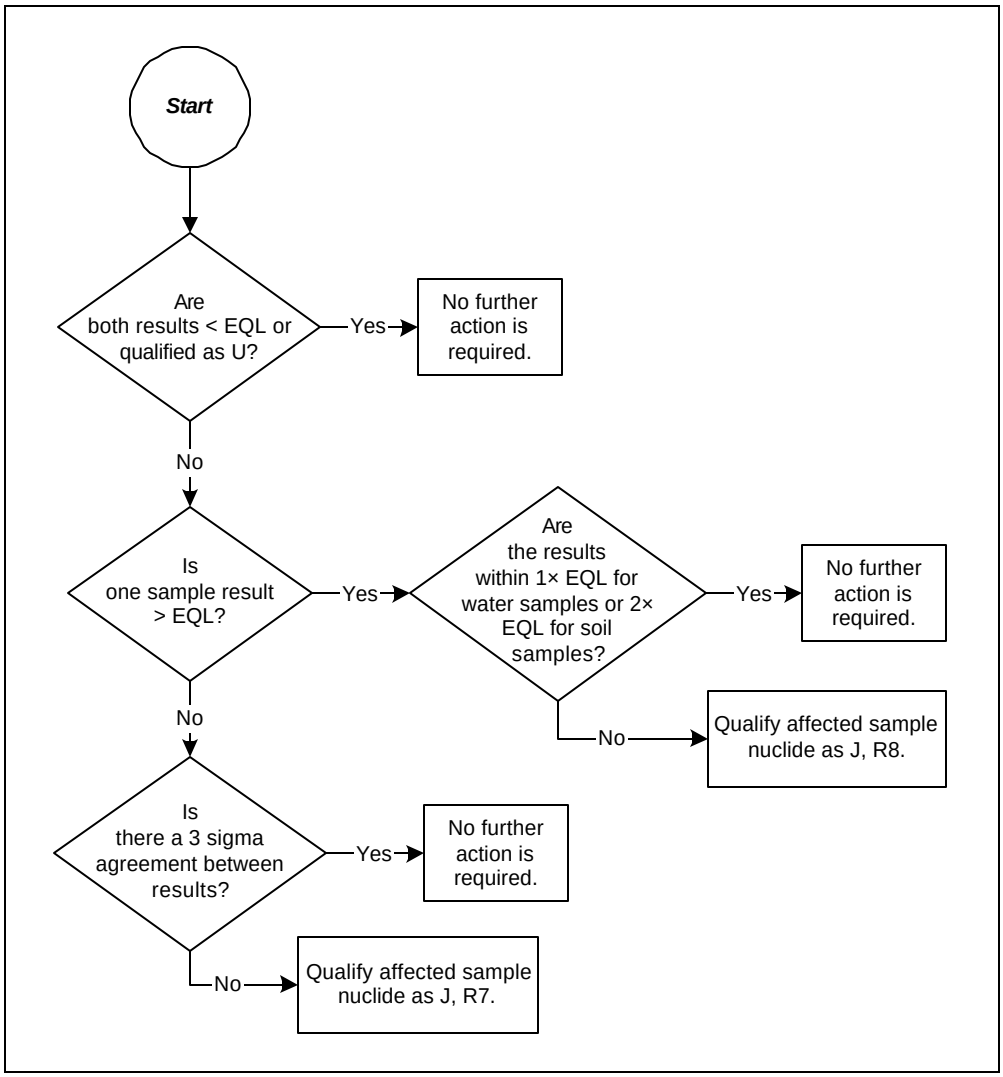

Figure 6.7-4. Logic diagram to determine applicable Laboratory qualifiers and reason codes for radiochemical analysis of duplicate sample results

6.7.5 EQL

6.7.5.1 Generally, radiochemical results are reported without any type of laboratory qualifier that indicated if results are nondetects. This test identifies nondetect radiochemical results. The test is performed by using either the minimum detectable activity (MDA) or decision level associated with the nuclide of interest. An MDA is a sample- and nuclide-specific number usually found in the raw data and not summarized on a form. The decision level result is associated with the blank and usually summarized on the blank form. If the validator cannot locate either an MDA or decision-level value in the data, he or she must contact the contract laboratory and request that they identify the location of the missing value. If no value for the either the MDA or decision level is listed in the data, the validator must request that the contract laboratory submit the missing value via FAX.

6.7.5.2 Use the following logic diagram (Figure 6.7-5) to determine which, if any, Laboratory qualifiers and reason codes are attached to the sample results.

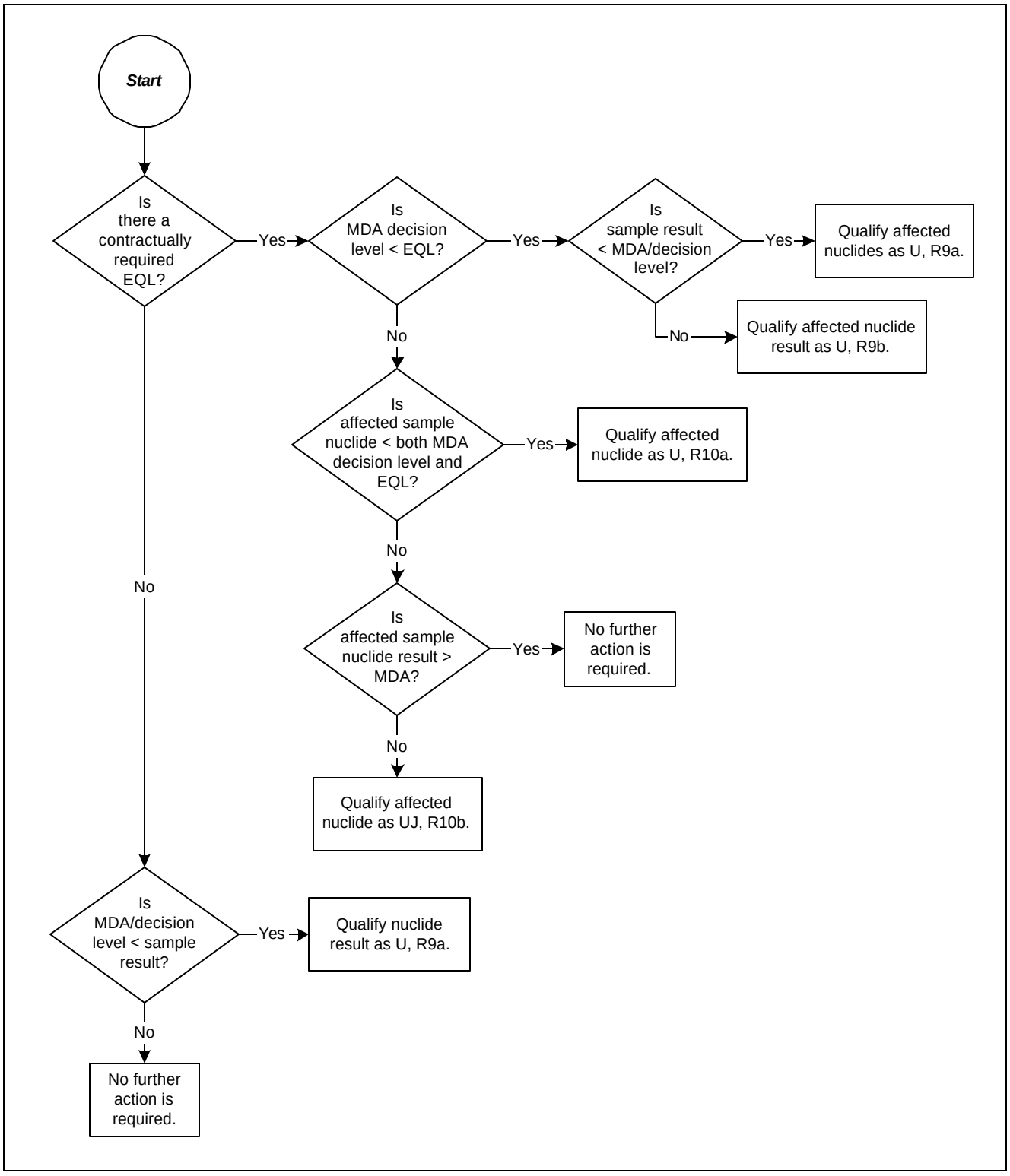

Figure 6.7-5. Logic diagram to determine applicable Laboratory qualifiers and reason codes for nondetected radiochemical results

6.7.6 Detection with Respect to the Reported Uncertainty

Due to time constraints, this important test is not done. Place an “NA” (for “not analyzed”) in the “Qualifiers Applied” column of the Reported Uncertainty section of the Baseline Validation Checklist for Radiochemistry Analysis (Attachment A).

6.7.7 Tracer/Carrier Recovery

The control criterion for the tracer recovery for alpha spectroscopy analysis is = 30%. The control criterion for the carrier recovery for beta/gamma analyses is = 40%.

6.7.7.1 This test is only applicable to alpha spectroscopy and beta/gamma analyses. The tracer/carrier recovery is a sample and nuclide specific result usually found in the raw data and not summarized on a form. If the validator cannot locate a tracer/carrier result, she or he must contact the contract laboratory and request that they identify the location of the tracer/carrier result. If no value for the tracer/carrier is listed in the data, the validator must request that the contract laboratory to submit the missing value via FAX.

6.7.7.2 Use the following logic diagram (Figure 6.7-6) to determine which, if any, Laboratory qualifiers and reason codes are attached to the sample results.

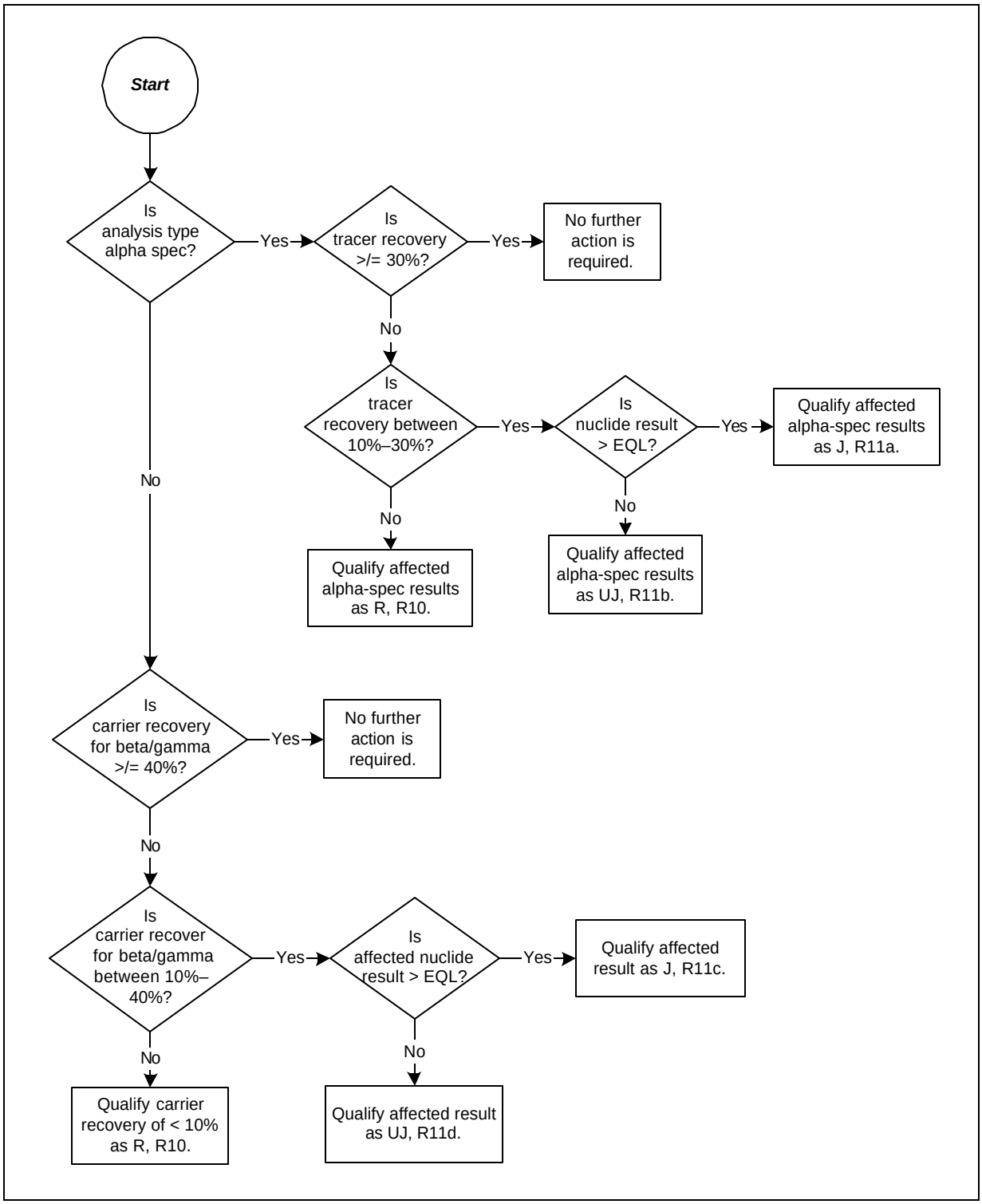

Figure 6.7-6. Logic diagram to determine applicable Laboratory qualifiers and reason codes for radiochemical tracer/carrier recovery results

6.7.8 Validation Completion

The **validator** is responsible for delivering the completed Baseline Validation Checklist(s) to the Sample Management Office (SMO) with the data package after the completion of the validation process.

7.0 REFERENCES

The following documents have been cited within this procedure.

AP-02.1, Procedure for LANL ER Records Management

QP-2.2, Personnel Orientation and Training

QP-4.2, Standard Operating Procedure Development

Los Alamos National Laboratory, July, 1995. “Environmental Restoration Project Statement of Work for Analytical Services,” RFP Number 9-SX1-Q4257, Revision 2, (Los Alamos National Laboratory, Los Alamos, New Mexico, 1995).

8.0 RECORDS

No records are generated as a result of this procedure.

9.0 ATTACHMENTS

The document user may employ documentation formats different from those attached to/named in this procedure—as long as the substituted formats in use provide, as a minimum, the information required in the official forms developed by the procedure.

Attachment A: Baseline Analytical Data Validation Cover Sheet and Checklists

Baseline Analytical Data Validation Cover Sheet

Section I.

Request Number: _____ Cost Code: _____

Validation Date: _____

Contract Labortatory Name: _____

Validator: _____ Organization: _____

Analytical Suite)s) (check all that apply):

- ☐ Volatile Organic
- ☐ Semivolatile Organic
- ☐ Organochlorine/Pesticide/Aroclor
- ☐ High Explosive
- ☐ Inorganic
- ☐ Radiochemistry

Other (describe): _____

Section II. Completeness Check

Yes	No	n/a	(check one)	Yes	No	n/a	(check one)
☐	☐	☐	1. Chain-of-Custody form(s)	☐	☐	☐	5. Standard Chromatagrams
☐	☐	☐	2. Case Narative	☐	☐	☐	6. Quantitation Reports
☐	☐	☐	3. Sample Results	☐	☐	☐	7. Quality Control Forms
☐	☐	☐	4. Sample Chromatagrams				

Identify any samples in the assigned Request Number that are missing:

_____ _____ _____ _____ _____ _____ _____ _____

_____ _____ _____ _____ _____ _____ _____ _____

Comments (include information about requests for further information submitted to the contract laboratory and agreed upon date of resolution and contract laboratory point of contact):

(Attach additional comment sheets as necessary)

Signature/1st validation: _____ Date: _____
(Print Name and Title, then Sign)

Signature/2nd validation: _____ Date: _____
(Print Name and Title, then Sign)

ER-SOP-15.17

Los Alamos
Environmental Restoration Project

Baseline Validation Checklist for Volatile Organic Analysis

Qualifiers Applied	Yes	No	Validation Criteria	Notes	Reason Code
	☐	☐	Is the instrument performance check (BFB) present?	Obtain from contract laboratory? — Qualifier = A	
	☐	☐	Is the initial calibration present? Comments:	Obtain from contract laboratory? — Qualifier = A On the Initial Calibration Form (CLP form VI), circle analytes that do not meet relative response factor (RRF) or % relative standard deviation (RSD) criteria.	
	☐	☐	Is the continuing calibration present? Comments:	Obtain from contract laboratory? — Qualifier = A On the Continuing Calibration Form (CLP form VII), circle analytes that do not meet RRF or % D (relative response factor difference) criteria.	
	☐	☐	Is the internal standards data present? Required internal standards: 1. chlorobenzene-d5 2. 1,4-difluorobenzene 3. 1,4-dichlorbenzene -d4 Retention time must be < ±30 sec. from previous continuing calibration. Response area must not vary by > a factor of 2 (+100%, -50%) from previous daily continuing calibration. Comments:	**Check internal standards manually during baseline validation.** Obtain internal standard data from contract laboratory? — Qualifier = A If internal standard retention time varies by > 30 seconds, qualify with PM, in which case focused validation checks the chromatographic profile for false positive or negative. If an internal standard area count is outside the -50%–+100% criterion, qualify positive results quantified by that internal standard as J, PM. Qualify nondetects quantified by the internal standard as UJ if area count < 50%. Very low or abrupt drop-off? — Qualifier = R Enter % variation in FIMAD table.	V1 V1a V2

Baseline Validation Checklist for Volatile Organic Analysis (continued)

Qualifiers Applied	Yes	No	Validation Criteria	Notes	Reason Code
	☐	☐	Are the system monitoring compounds (SMCs or surrogates) present? **Water Recovery Criteria** Compound — Recovery Toluene-d8 — 88%–110% BFB — 86%–115% Dibromofluoromethane — 86%–118% **Soil Recovery Criteria** Compound — Recovery Toluene-d8 — 81%–117% BFB — 74%–121% Dibromofluoromethane — 80%–120% Comments:	Obtain from contract laboratory? — Qualifier = A If the SMC % recovery > the upper limit, qualify detected target compounds as J+. If the SMC % recovery > the upper limit, nondetected target compounds are not qualified. If the SMC % recovery < the lower limit but > 10%, qualify detected compounds as J-. For detected compounds, use the same J- qualifier if the SMC recovery < 10%. For nondetected target compounds, the quantitation limit is qualified as approximated, UJ, when the SMC recovery = 10%. For nondetected analytes, if the SMC recovery < 10%, reject R, PM. Do not qualify results for diluted samples based on SMC recoveries. Enter % recovery in FIMAD table.	V3 V3a V3b V3c V3d

Baseline Validation Checklist for Volatile Organic Analysis (concluded)

Qualifiers Applied	Yes	No	Validation Criteria	Notes	Reason Code
	☐	☐	Are the method blanks present? Separate blank for each method, matrix, and/or 12-hour batch. Target compounds must be = the EQL except acetone, methylene chloride, and 2-butanone which can be present at = 5× the EQL. Comments:	Obtain from contract laboratory? — Qualifier = A If target compound is found in blank but not in sample, add no qualifier. If the sample result > the EQL and < 5× (< 10× for noted exceptions) multiple of blank, elevate the EQL to sample result and qualify as U. If the sample result < the EQL and < 5× (< 10× for noted exceptions) multiple of blank, elevate sample results to the EQL and qualify as U. If the sample result > 5× multiple, OK	V4 V5
	☐	☐	Was sample analyzed within its holding time? Analysis within 14 days of sample collection for soil samples and within 7 days for water samples. Comments:	Compare the date of analysis with the sampling date on the Analytical Request. Qualifier = PM if holding time was not met.	
	☐	☐	Are tentatively identified compounds (TICs) present (if requested)? Comments:		

Definitions of Laboratory Qualifiers and Laboratory Reason Codes for Volatile Organic Analysis

Laboratory Validation Qualifiers

U The analyte was analyzed for but not detected above the reported EQL.

J The analyte was positively identified, the associated numerical value is the approximate concentration of the analyte in the sample:
J+ = likely to have a high bias,
J- = likely to have a low bias.

UJ The analyte was analyzed for but not detected. The associated value is an estimate.

R The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality-control criteria. Presence or absence cannot be verified.

Note: Any results qualified as “R” should be looked at for relevance for data use. Thus, “R” implies “PM” also, and must not be used alone.

P Use professional judgment based on data use. It usually has an “M” with it, which indicates that a manual check should be made if the data that is qualified with the “P” is important to the data user.

In addition, “PM” also means that a decision must be made by the project manager or a delegate with regard to the need for further review of the data. This review should include some consideration of potential impact that could result from using the “P” qualified data. (For example, in the case of holding-time exceedance, the project manager or delegate can decide to use the data with no qualification when analytes of interest are known to not be adversely affected by holding-time exceedances. Another example is the case where soil sample duplicate analyses for metals exceed the precision criteria. Because this is likely due to sample nonhomogeneity rather than contract laboratory error, the manager or delegate must decide how to use the data.)

PM Manual review of raw data is recommended in order to determine if the defect impacts data use, as in “R” above.

Laboratory Reason Codes for Validation Qualifiers for Volatile Organic Analysis

V0 The internal standard retention time has shifted by more than ±30 seconds, which could affect compound identification and cause false-positive or -negative results.

V1 The internal standard area count for the quantitating internal standard is outside the -50%, +100% window in relation to the previous continuing calibration, which could affect the accuracy of the quantitation of the associated analytes and the correct quantitation of system monitoring compound (SMC—often called “surrogates”) recoveries.

V1a Nondetected results; and the area count for the quantitating internal standard is less than 50% of the area count for the previous continuing calibration, which greatly increases the potential for false-negative results.

V2 Very low or abrupt drop-off of quantitating internal standard, which indicates an increased potential for false-negative results and possibly other problems with sample quantitation.

V3 The surrogate percent recovery is greater than the upper limit, which indicates a potential high bias in the results and a potential for false-positive results.

V3a The surrogate percent recovery is less than the lower limit *but* is greater than 10%, which indicates a potential low bias in the results.

V3b The surrogate recovery is less than 10%, which indicates an increased potential for a low bias in the results.

V3c Indicates that the quantitation limit is approximated for nondetects because of surrogate recovery that is less than the lower limit but is greater than 10%, which also indicates an increased potential for false-negative results.

V3d Surrogate percent recovery that is less than 10% and a result that is a nondetect, which indicates a greatly increased potential for false-negative results.

V4 A sample result that is greater than the estimated quantitation limit (EQL) *and* is less than 5 times (10 times for acetone, methylene chloride, and 2-butanone) the concentration of the related analyte in the blank, which indicates the reported detection is considered to be indistinguishable from blank contamination.

V5 A sample result that is less than the EQL *and* is less than 5 times (and 10 times for acetone, methylene chloride, and 2-butanone) the concentration of the related analyte in the blank, which indicates the detected result was indistinguishable from blank contamination and the detected result was changed to nondetected at the EQL.

V5a A sample result that is greater than the EQL *and* is greater than 5 times (10 times for acetone, methylene chloride, and 2-butanone) the concentration of the

analyte in the blank, which indicates the reported result is not likely to be related to the contamination in the associated blank.

V6 The response factor for the analytes is less than 0.05, which indicates a sensitivity problem that could prohibit analyte detection if present at low concentrations.

V7 The percent RSD or percent D exceeds the specification which may affect quantitation, which indicates potential quantitation problems in the analyses.

V8 Instrument Performance Check has ion ratios out of specification, which may affect compound identification.

V9 Holding time is exceeded. An evaluation of the data of interest with respect to holding-time-exceedance impact (technically) is recommended. Factors to consider include sample preservation; sample-storage practices; use of the data; levels of contamination found in the sample; and the physical, chemical, and biological stability of the target analytes in the sample matrix.

V10 The result is higher than the high point of the calibration (or outside the linear range). This usually results in a negative bias in the reported concentration.

Volatile Organic Analysis
Sample Analytical Results

Volatile Organic Analysis
QC Results

Baseline Validation Checklist for Semivolatile Organic Analysis

Qualifiers Applied	Yes	No	Validation Criteria	Notes	Reason Code
	☐	☐	Is the instrument performance check (DFTPP) present?	Obtain from contract laboratory? — Qualifier = A	
	☐	☐	Is the initial calibration present? Comments:	Obtain from contract laboratory? — Qualifier = A On the Initial Calibration Form (CLP form VI), circle analytes that do not meet relative response factor (RRF) or % relative standard deviation (RSD) criteria.	
	☐	☐	Is the continuing calibration present? Comments:	Obtain from contract laboratory? — Qualifier = A On the Continuing Calibration Form (CLP form VII), circle analytes that do not meet RRF or % D (relative response factor difference) criteria.	
	☐	☐	Is the internal standards data present? Required internal standards: 1. 1,4-dichlorobenzene-d4 4. phenanthrene-d10 2. naphthalene-d8 5. chrysene-d12 3. acenaphthene-d10 6. perylene-d12 Retention time must be < ±30 sec. from previous continuing calibration. Response area must not vary by > a factor of 2 (+100%, -50%) from previous daily continuing calibration. Comments:	**Check internal standards manually during baseline validation.** Obtain internal standard data from contract laboratory? — Qualifier = A If internal standard retention time varies by > 30 seconds, qualify with PM, in which case focused validation checks the chromatographic profile for false positive or negative. If an internal standard area count is outside the -50%–+100% criterion, qualify positive results quantified by that internal standard as J, PM. Qualify nondetects quantified by the internal standard as UJ if the area count < 50%. Very low or abrupt drop-off? — Qualifier = R Enter % variation in FIMAD table.	SV1 SV1a SV2

Baseline Validation Checklist for Semivolatile Organic Analysis (continued)

Qualifiers Applied	Yes	No	Validation Criteria	Notes	Reason Code
	☐	☐	Are the system monitoring compounds (SMCs or surrogates) present? **Water Recovery Criteria** Compound — Recovery Nitrobenzene-d5 35%–114% 2-Fluorobiphenyl 43%–116% p-Terphenyl 33%–141% Phenol-d6 10%–94% 2-Fluorophenol 21%–100% 2,4,6-Tribromophenol 10%–123% **Soil Recovery Criteria** Compound — Recovery Nitrobenzene-d5 23%–120% 2-Fluorobiphenyl 30%–115% p-Terphenyl 18%–137% Phenol-d6 24%–113% 2-Fluorophenol 25%–121% 2,4,6-Tribromophenol 19%–122% Comments:	Obtain from contract laboratory? — Qualifier = A If 2 or more surrogates in either semivolatile fraction have a recovery of > the upper limit, specify the fraction being qualified and qualify detected target compounds with J+ and no qualifier for nondetects. If 2 or more surrogates in either fraction have a recovery of > 10% but < the lower limit, specify the fraction being qualified and qualify as J-. Qualify nondetected target compounds as UJ. If > 2 surrogates are out in either fraction, one with a recovery of > the upper limit and one with a recovery of > 10% but < the lower limit, qualify as U, J. If any surrogate in either fraction shows < 10% recovery, specify the fraction being qualified and qualify detected compounds as J-. If any surrogate in either fraction < 10% recovery, specify the fraction being qualified and qualify nondetects as R, PM. Do not qualify results for diluted samples based on surrogate recoveries. Enter % recovery in FIMAD table.	SV3 SV3a SV3c SV3e SV3b SV3d

Baseline Validation Checklist for Semivolatile Organic Analysis (concluded)

Qualifiers Applied	Yes	No	Validation Criteria	Notes	Reason Code
	☐	☐	Are the method blanks present? Separate blank for each method, matrix, and/or 12-hour batch. Target compounds must be = the EQL except acetone, methylene chloride, and 2-butanone which can be present at = 5× the EQL. Comments:	Obtain from contract laboratory? — Qualifier = A If target compound found in blank but not in sample, add no qualifier. If the sample result > the EQL and < 5× (< 10× for noted exceptions) multiple of blank, elevate the EQL to sample result and qualify as U. If the sample result < the EQL and < 5× (< 10× for noted exceptions) multiple of blank, elevate sample results to the EQL and qualify as U. If the sample result > 5× multiple, OK	SV4 SV5
	☐	☐	Was sample analyzed within its holding time? Extraction within 14 days of sample collection for soil samples and within 7 days for water samples. Then, analysis within 40 days of extraction Comments:	Compare the date of extraction with the sampling date on the Analytical Request and the date of analysis with the date of extraction. Qualifier = PM if holding time was not met.	
	☐	☐	Are tentatively identified compounds (TICs) present (if requested)? Comments:		

Definitions of Laboratory Qualifiers and Laboratory Reason Codes for Semivolatile Organic Analysis

Laboratory Validation Qualifiers

U The analyte was analyzed for but not detected above the reported EQL.

J The analyte was positively identified, the associated numerical value is the approximate concentration of the analyte in the sample:
J+ = likely to have a high bias,
J- = likely to have a low bias.

UJ The analyte was analyzed for but not detected. The associated value is an estimate.

R The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality-control criteria. Presence or absence cannot be verified.

Note: Any results qualified as “R” should be looked at for relevance for data use. Thus, “R” implies “PM” also, and must not be used alone.

P Use professional judgment based on data use. It usually has an “M” with it, which indicates that a manual check should be made if the data that is qualified with the “P” is important to the data user.

In addition, “PM” also means that a decision must be made by the project manager or a delegate with regard to the need for further review of the data. This review should include some consideration of potential impact that could result from using the “P” qualified data. (For example, in the case of holding-time exceedance, the project manager or delegate can decide to use the data with no qualification when analytes of interest are known to not be adversely affected by holding-time exceedances. Another example is the case where soil sample duplicate analyses for metals exceed the precision criteria. Because this is likely due to sample nonhomogeneity rather than contract laboratory error, the manager or delegate must decide how to use the data.)

PM Manual review of raw data is recommended in order to determine if the defect impacts data use, as in “R” above.

Laboratory Reason Codes for Validation Qualifiers for Semivolatile Organic Analysis

SV0 The internal standard retention time has shifted by more than ±30 seconds, which could affect compound identification and cause false positives or negatives.

SV1 The internal standard area count for the quantitating internal standard is outside the -50%, +100 % window in relation to the previous continuing calibration, which could affect the accuracy of the quantitation of the associated analytes and the correct quantitation of system monitoring compound (SMC—often called “surrogates”) recoveries.

SV1a Nondetected results; and the area count for the quantitating internal standard is less than 50% of the area count for the previous continuing calibration, which greatly increases the potential for false-negative results.

SV2 Very low or abrupt drop-off of quantitating internal standard, which indicates an increased potential for false-negative results and possibly other problems with sample quantitation.

SV3 Two or more surrogates in either semivolatile fraction have a percent recovery that is greater than the upper limit, which indicates a potential high bias in the results and a potential for false-positive results.

SV3a Two or more surrogates in either semivolatile fraction have a percent recovery that is greater than 10% *but* that is less than the lower limit, which indicates a potential low bias in the results.

SV3b A surrogate in the related semivolatile fraction has a recovery of less than 10%, which indicates an increased potential low bias in the results.

SV3c Result is a nondetect and two or more surrogates have a recovery of greater than 10% but less than the lower limit, which indicates an increased potential for false-negative results.

SV3d Result is a nondetect and a surrogate in the related semivolatile fraction has a percent recovery of less than 10%, which indicates a greatly increased potential for false-negative results.

SV3e Recovery of one surrogate in a fraction is greater than the upper limit *and* one is less than the lower limit but is grater than 10%, which indicates a potential bias in the results, however, the direction of the bias is uncertain.

SV4 The sample result is greater than the estimated quantitation limit (EQL) *and* is less than 5 times (10 times for common phthalates) the concentration of the related analyte in the blank, which indicates the reported detection is considered to be indistinguishable from blank contamination.

SV5 The sample result is less than the EQL *and* is less than 5 times (10 times for common phthalates) the concentration of the analyte in the blank, which

indicates the detected result was indistinguishable from blank contamination and the detected result was changed to nondetected at the EQL.

SV5a The sample result is greater than the EQL *and* is greater than 5 times (10 times for common phthalates) the concentration of the analyte in the blank, which indicates the reported result is not likely to be related to the contamination in the associated blank.

SV6 The response factor for the analytes is less than 0.05, which indicates a sensitivity problem that could prohibit detection if present at low concentrations

SV7 The percent RSD or percent D exceeds the specification, which may affect quantitation, which indicates potential quantitation problems in the analyses.

SV8 Instrument Performance Check has “important” ion(s) or ion ratios out of specification, which may affect compound identification.

SV9 Holding time is exceeded. An evaluation of the data of interest with respect to holding-time-exceedance impact (technically) is recommended. Factors to consider include sample preservation; sample-storage practices; use of the data; levels of contamination found in the sample; and the physical, chemical, and biological stability of the target analytes in the sample matrix.

SV10 The result is higher than the high point of the calibration (or outside the linear range). This usually results in a negative bias in the reported concentration.

Semivolatile Organic Analysis
Sample Analytical Results

Semivolatile Organic Analysis
QC Results

Baseline Validation Checklist for Organochlorine/Pesticide/Aroclor Analysis

Qualifiers Applied	Yes	No	Validation Criteria	Notes	Reason Code
	☐	☐	Is the initial calibration present? Comments:	Obtain from contract laboratory? — Qualifier = A	
	☐	☐	Is the daily calibration verification present? Comments:	Obtain from contract laboratory? — Qualifier = A	
	☐	☐	Are the system monitoring compounds (SMCs or surrogates) present? **Recovery Criteria** Compound — Recovery Nitrobenzene-d5 — 50%–160% 2,4,6-Tribromophenol — 50%–160% Comments:	Obtain from contract laboratory? — Qualifier = A If either surrogate's % recovery > the upper limit, qualify detected target compounds as J+. If either surrogate's % recovery ≥ 10% but ≤ the lower limit, qualify detected compounds as J-. If either surrogate's % recovery is ≥ 10% but ≤ the lower limit, qualify nondetected target analytes as UJ. If either surrogate's % recovery < 10%, qualify detected compounds as J-. If either surrogate's % recovery < 10%, qualify nondetects as RPM. Enter % recovery in FIMAD table.	P3 P3a P3c P3b P3d

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Baseline Validation Checklist for Organochlorine/Pesticide/Aroclor Analysis (continued)

Qualifiers Applied	Yes	No	Validation Criteria	Notes	Reason Code
	☐	☐	Is retention-time windows data present? Surrogates' retention times should not shift by > 0.05 min. Comments:	Obtain from contract laboratory? — Qualifier = A If either of the surrogate's retention time shifts by > ±0.05 minutes, qualify the results as PM. (An examination of chromatographic peaks will be required to determine if target analytes are present.)	
	☐	☐	Are the method blanks present? Separate blank for each method, matrix, and/or analytical batch. Target analytes must be < the EQL. Comments:	Obtain from contract laboratory? — Qualifier = A If target compound is found in blank but not in sample, add no qualifier. If the sample result > the EQL and < 5× multiple of blank, elevate the EQL to sample result and qualify as U. If the sample result < the EQL and < 5× multiple of blank, elevate sample results to the EQL and qualify as U. If the sample result > 5× multiple, OK	P4 P5
	☐	☐	Is the breakdown data present? Comments:	Obtain from contract laboratory? — Qualifier = A The breakdown criteria are = 20% for either 4,4'-DDT or endrin -or- = 30% for combined breakdown Qualifier = P	

Baseline Validation Checklist for Organochlorine/Pesticide/Aroclor Analysis (concluded)

	☐	☐	Was sample analyzed within its holding time? Extraction within 14 days of sample collection for soil samples and within 7 days for water samples. Then, analysis within 40 days of extraction Comments:	Compare the date of extraction with the sampling date on the Analytical Request and the date of analysis with the date of extraction. Qualifier = PM if holding time was not met.	

Definitions of Laboratory Qualifiers and Laboratory Reason Codes for Organochlorine/Pesticide/Aroclor Analysis

Laboratory Validation Qualifiers

U The analyte was analyzed for but not detected above the reported EQL.

J The analyte was positively identified, the associated numerical value is the approximate concentration of the analyte in the sample:
J+ = likely to have a high bias,
J- = likely to have a low bias.

UJ The analyte was analyzed for but not detected. The associated value is an estimate.

R The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality-control criteria. Presence or absence cannot be verified.

Note: Any results qualified as “R” should be looked at for relevance for data use. Thus, “R” implies “PM” also, and must not be used alone.

P Use professional judgment based on data use. It usually has an “M” with it, which indicates that a manual check should be made if the data that is qualified with the “P” is important to the data user.

In addition, “PM” also means that a decision must be made by the project manager or a delegate with regard to the need for further review of the data. This review should include some consideration of potential impact that could result from using the “P” qualified data. (For example, in the case of holding-time exceedance, the project manager or delegate can decide to use the data with no qualification when analytes of interest are known to not be adversely affected by holding-time exceedances. Another example is the case where soil sample duplicate analyses for metals exceed the precision criteria. Because this is likely due to sample nonhomogeneity rather than contract laboratory error, the manager or delegate must decide how to use the data.)

PM Manual review of raw data is recommended in order to determine if the defect impacts data use, as in “R” above.

Laboratory Reason Codes for Validation Qualifiers
for Organochlorine/Pesticide/Aroclor Analysis

P3 The system monitoring compound (SMC—often called “surrogates”) percent recovery is greater than the upper limit, which indicates a potential high bias in the results and a potential for false-positive results.

P3a The surrogate percent recovery is greater than 10% *but* is less than the lower limit, which indicates a potential low bias in the results.

P3b The surrogate percent recovery is less than 10%, which indicates an increased potential low bias in the results.

P3c Result is a nondetect and the surrogate percent recovery is greater than 10% *but* is less than the lower limit, which indicates a potential for false-negative results.

P3d Result is a nondetect and the surrogate percent recovery is less than 10%, which indicates a greatly increased potential for false-negative results.

P4 The sample result is greater than the estimated quantitation limit (EQL) *and* is less than 5 times the concentration of the related analyte in the blank, which indicates the reported detection is considered to be indistinguishable from blank contamination.

P5 The sample result is less than the EQL *and* is less than 5 times the concentration of the analyte in the blank, which indicates the detected result was indistinguishable from blank contamination and the detected result was changed to nondetected at the EQL.

P5a The sample result is greater than the EQL *and* is greater than 5 times the concentration of the analyte in the blank, which indicates the reported result is not likely to be related to the contamination in the associated blank.

P6 The percent RSD or percent D exceeds the specification—apply reason code to a positive result, which indicates potential quantitation problems in the analyses and the potential for false-positive results.

P7 The percent RSD or percent D exceeds the specification—apply reason code to a nondetect, which indicates potential quantitation problems in the analyses and the potential false-negative results.

P8 Analyte concentration exceeds linear range and sample was not diluted to within that range. This usually results in a negative bias in the reported concentration.

P9 Holding time is exceeded. An evaluation of the data of interest with respect to holding-time exceedance impact (technically) is recommended. Factors to consider include sample preservation; sample-storage practices; use of the data; levels of contamination found in the sample; and the physical, chemical, and biological stability of the target analytes in the sample matrix.

P10 Breakdown criteria have been exceeded, this indicates poor instrument performance which can result in a low bias in the reported results and potential false-negative results for labile compounds and potential false-positive results for breakdown products.

P11 Surrogate retention time has shifted by more than 0.05 minutes, possibly affecting analyte identification and causing false positives or negatives.

Organochlorine/Pesticide/Aroclor Analysis
Sample Analytical Results

Organochlorine/Pesticide/Aroclor Analysis QC Results

Baseline Validation Checklist for High Explosives Analysis

Qualifiers Applied	Yes	No	Validation Criteria	Notes	Reason Code
	☐	☐	Is the initial calibration present? Comments:	Obtain from contract laboratory? — Qualifier = A	
	☐	☐	Is the daily calibration present? Comments:	Obtain from contract laboratory? — Qualifier = A	
	☐	☐	Is the laboratory control sample (LCS) present? 60%–120% recovery of analytes (advisory). Comments:	Obtain from contract laboratory? — Qualifier = A If criterion is not met, qualify each analyte associated with the LCS in the same batch as J- for a recovery of < 60%. If criterion is not met, qualify each analyte associated with the LCS in the same batch as J+ for a recovery of > 120%. For 0% recovery, qualify nondetects as RPM.	H6a H6 H6d
	☐	☐	Are the system monitoring compounds (SMCs or surrogates) present? Target compounds: 3,4-Dinitrotoluene (required) and 4-Nitroaniline (optional). Comments:	Obtain from contract laboratory? — Qualifier = A Enter % recovery in FIMAD table.	

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Baseline Validation Checklist for High Explosives Analysis (concluded)

Qualifiers Applied	Yes	No	Validation Criteria	Notes	Reason Code
	☐	☐	Are the method blanks present? Target analytes must be = the EQL. Comments:	Obtain from contract laboratory? — Qualifier = A If target compound is found in blank but not in sample, add no qualifier. If the sample result > the EQL and < 5× multiple of blank, elevate the EQL to sample result and qualify as U. If the sample result < the EQL and < 5× multiple of blank, elevate sample results to the EQL and qualify as U. If the sample result > 5× multiple, OK	 H4 H5
	☐	☐	Was sample analyzed within its holding time? Extraction within 14 days of sample collection for soil samples and within 7 days for water samples. Then, analysis within 40 days of extraction Comments:	Compare the date of extraction with the sampling date on the Analytical Request and the date of analysis with the date of extraction. Qualifier = PM if holding time was not met.	

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Definitions of Laboratory Qualifiers and Laboratory Reason Codes for High Explosives Analysis

Laboratory Validation Qualifiers

U The analyte was analyzed for but not detected above the reported EQL.

J The analyte was positively identified, the associated numerical value is the approximate concentration of the analyte in the sample:
J+ = likely to have a high bias,
J- = likely to have a low bias.

UJ The analyte was analyzed for but not detected. The associated value is an estimate.

R The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality-control criteria. Presence or absence cannot be verified.

Note: Any results qualified as “R” should be looked at for relevance for data use. Thus, “R” implies “PM” also, and must not be used alone.

P Use professional judgment based on data use. It usually has an “M” with it, which indicates that a manual check should be made if the data that is qualified with the “P” is important to the data user.

In addition, “PM” also means that a decision must be made by the project manager or a delegate with regard to the need for further review of the data. This review should include some consideration of potential impact that could result from using the “P” qualified data. (For example, in the case of holding-time exceedance, the project manager or delegate can decide to use the data with no qualification when analytes of interest are known to not be adversely affected by holding-time exceedances. Another example is the case where soil sample duplicate analyses for metals exceed the precision criteria. Because this is likely due to sample nonhomogeneity rather than contract laboratory error, the manager or delegate must decide how to use the data.)

PM Manual review of raw data is recommended in order to determine if the defect impacts data use, as in “R” above.

Laboratory Reason Codes for Validation Qualifiers for High Explosives Analysis

H4 The sample result is greater than the estimated quantitation limit (EQL) *and* is less than 5 times the concentration of the related analyte in the blank, which indicates the reported detection is considered to be indistinguishable from blank contamination.

H5 The sample result is less than the EQL *and* is less than 5 times the concentration of the analyte in the blank, which indicates the detected result was indistinguishable from blank contamination and the detected result was changed to nondetected at the EQL.

H5a The sample result is greater than the EQL *and* is greater than 5 times the concentration of the analyte in the blank, which indicates the reported result is not likely to be related to the contamination in the associated blank.

H6 The recovery of system monitoring compounds (SMCs—often called "surrogates") or analyte in the laboratory control sample (LCS) is greater than the upper limit, which indicates a potential high bias in the results and a potential for false-positive results.

H6a The recovery of surrogates or analyte in the LCS is less than the lower limit, which indicates a potential low bias in the results.

H6d The result is a nondetect and the recovery of surrogates or the analyte in the LCS is less than the lower limit, which indicates a potential for false-negative results.

H7 The percent RSD or the percent D exceeds specification—apply reason code to a positive result, which indicates potential quantitation problems in the analyses and the potential for false-positive results.

H8 The percent RSD or the percent D exceeds the specification—apply reason code to a nondetect, which indicates potential quantitation problems in the analyses and the potential false-negative results.

H9 Holding time is exceeded. An evaluation of the data of interest with respect to holding-time exceedance impact (technically) is recommended. Factors to consider include sample preservation; sample storage practices; use of the data; levels of contamination found in the sample; and the physical, chemical, and biological stability of the target analytes in the sample matrix.

H10 Duplicate RPD is greater than the advisory 20% limit.

High Explosives Analysis
Sample Analytical Results

High Explosives Analysis
QC Results

Baseline Validation Checklist for Inorganic Analysis

Qualifiers Applied	Yes	No	Validation Criteria	Notes	Reason Code
	☐	☐	Is the initial calibration present? Comments:	Obtain from contract laboratory? — Qualifier = A	
	☐	☐	Is the continuing calibration present? Comments:	Obtain from contract laboratory? — Qualifier = A	
	☐	☐	Is the initial-calibration blank present? Comments:	Obtain from contract laboratory? — Qualifier = A Use "worse" case blank. Sample results > the EDL but < 5× amount found in the blank should be qualified as U. Analyze the blank at each wavelength.	I4
	☐	☐	Is the continuing-calibration blank present? Comments:	Analyze a blank at the beginning and end of each run. Initial- and continuing-calibration blank analyzed at a frequency of 10% or 2 hours. If blank results > the EDL re-analyze previous 10 samples.	
	☐	☐	Is the preparation blank present? Comments:	<u>Preparation blank</u> Analyze one per batch. Blank results must be ≤ the EDL, or if concentration of analyte < 10× the EDL, repeat all samples in batch.	

Baseline Validation Checklist for Inorganic Analysis (continued)

Qualifiers Applied	Yes	No	Validation Criteria	Notes	Reason Code
	☐	☐	Is the ICP interference-check sample (ICS) present? Solution contains: Ag, Ba, Be, Cd, Co, Cr, Cu, Mn, Ni, Pb, V, Zn. ICS results must be ±20% of true value. Comments:	Obtain the ICS from contract laboratory? — Qualifier = A Analyze the ICS at the beginning of each run. Analyze each wavelength used for samples. If the ICS recovery > 120% and the results are > the EDL, qualify data as J+ If the ICS recovery is between 50%–79%, qualify data as J-. If the ICS recovery < 50% and results are < the EDL, qualify results as RPM.	 I7 I7a I7b
	☐	☐	Is the spike sample analysis present? One spike sample per run, per matrix, per concentration, per sample delivery group (SDG) % recovery: 75%–125% (unless conc. > 4× spike conc.) Comments:	Obtain from contract laboratory? — Qualifier = A If spike recovery > 125% and results < the EDL, OK If spike recovery < 75% and results > the EDL, qualify results as J- if same matrix, as P if not. If spike recovery > 125% and results > the EDL, qualify data as J+ if same matrix, as P if not. If spike recovery is between 30%–74% and sample results < the EDL, qualify as UJ if same matrix, as P if not. If spike recovery results < 30% and results < the EDL, qualify as RPM.	 I3a I3 I3e I3d

Baseline Validation Checklist for Inorganic Analysis (concluded)

Qualifiers Applied	Yes	No	Validation Criteria	Notes	Reason Code
	☐	☐	Are the duplicates present? One per batch of sample of like matrix for each SDG. Relative % difference (RPD) ≤ 20% (advisory). If either the sample or duplicate value is less than 5× the EDL, there is a control limit of ± the EDL. Comments:	Obtain from contract laboratory? — Qualifier = A Qualifier = P because of nature of Laboratory samples.	
	☐	☐	Is the laboratory control sample (LCS) present? Aqueous LCS per batch/SDG. 80%–120% recovery of analytes (except Ag and Sb). Comments:	Obtain from contract laboratory? — Qualifier = A If criterion is not met, qualify each analyte associated with the LCS in the same batch as J- for a recovery of < 80%. If criterion is not met, qualify each analyte associated with the LCS in the same batch as J+ for a recovery of > 120%.	I6a I6
	☐	☐	Was sample analyzed within its holding time? Analyze water samples for mercury within 28 days of sample collection. Analyze water samples for cyanides within 14 days of sample collection. Comments:	Compare the date of extraction with the sampling date on the Analytical Request. Qualifier = PM if holding time was not met.	

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Definitions of Laboratory Qualifiers and Laboratory Reason Codes for Inorganic Analysis

Laboratory Validation Qualifiers

U The analyte was analyzed for but not detected above the reported EQL.

J The analyte was positively identified, the associated numerical value is the approximate concentration of the analyte in the sample:
J+ = likely to have a high bias,
J- = likely to have a low bias.

UJ The analyte was analyzed for but not detected. The associated value is an estimate.

R The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality-control criteria. Presence or absence cannot be verified.

Note: Any results qualified as “R” should be looked at for relevance for data use. Thus, “R” implies “PM” also, and must not be used alone.

P Use professional judgment based on data use. It usually has an “M” with it, which indicates that a manual check should be made if the data that is qualified with the “P” is important to the data user.

In addition, “PM” also means that a decision must be made by the project manager or a delegate with regard to the need for further review of the data. This review should include some consideration of potential impact that could result from using the “P” qualified data. (For example, in the case of holding-time exceedance, the project manager or delegate can decide to use the data with no qualification when analytes of interest are known to not be adversely affected by holding-time exceedances. Another example is the case where soil sample duplicate analyses for metals exceed the precision criteria. Because this is likely due to sample nonhomogeneity rather than contract laboratory error, the manager or delegate must decide how to use the data.)

PM Manual review of raw data is recommended in order to determine if the defect impacts data use, as in “R” above.

Laboratory Reason Codes for Validation Qualifiers for Inorganic Analysis

I3 Spike recovery is greater than the upper limit (125%) *and* the sample results are greater than the estimated detection limit (EDL).

I3a Spike recovery is less than the lower limit (75%) *and* the sample results are greater than the EDL.

I3d Spike recovery is less than 30% *and* the sample results are less than the EDL.

I3e Spike recovery is between 30%–74% and the sample results are less than the EDL.

I4 Using the worse-case blank, the sample results are greater than the EDL but are less than five times the concentration of the related analyte in the blank.

I6 Recovery of analyte in the laboratory control sample (LCS) is greater than the upper limit.

I6a Recovery of analyte in the LCS is less than the lower limit.

I7 Interference-check sample (ICS) recovery is greater than 120% and the sample results are greater than the EDL.

I7a ICS recovery is between 50%–79%.

I7b ICS recovery is less than 50% *and* result is less than the EDL.

I8a For initial calibration verification (ICV) and continuing calibration verification (CCV), recovery is lower than specification.

I8b For ICV and CCV, recovery is higher than specification.

I8c For ICV and CCV, results are positive but are less than the method detection limit (MDL) *and* recovery is lower than the specification.

I8d For ICV and CCV, recovery is extremely low and detection may be impaired.

I8e For ICV and CCV, recovery is abnormally high—apply to results that are greater than the MDL.

I9 Holding time for mercury has been exceeded. Positive results may be biased low and nondetects may potentially be false negatives.

Inorganic Analysis
Sample Analytical Results

Inorganic Analysis
QC Results

Baseline Validation Checklist for Radiochemistry Analysis

Qualifiers Applied	Yes	No	Validation Criteria	Notes	Reason Code
	☐	☐	Is the laboratory control sample (LCS) present? 1 per sample delivery group (SDG). 75%–125% recovery. Comments:	Obtain from contract laboratory? — Qualifier = A If criterion is not met, qualify each analyte associated with the LCS in the same batch as J- for a recovery of < 75%. If criterion is not met, qualify each analyte associated with the LCS in the same batch as J+ for a recovery of > 125%. If the LCS sample tracer/carrier recovery is < 10%, qualify the associated sample data as P for nondetects.	R6a R6 R6d
	☐	☐	Is the matrix spike present? 1 per SDG. 75%–125% recovery. **Note:** As opposed to other tests, the matrix spike test will qualify samples on the basis of their connection by SDG and not by analytical batch. Comments:	Obtain from contract laboratory? — Qualifier = A If criterion is not met, qualify all analytes associated with the matrix spike in the same batch as J- for a recovery of < 75% if all samples in batch are of same matrix. If criterion is not met, qualify all analytes associated with the matrix spike in the same batch as J+ for a recovery of > 125% if all samples in batch are of same matrix. If there is variation of matrix, apply the qualifier P for samples not of same matrix as spiked sample.	R3a R3 R3b
	☐	☐	Is the method blank present? 1 per SDG. Target analytes must be = 5× the amount in the blank. Comments:	Obtain from contract laboratory? — Qualifier = A If the sample result > the EQL, or no EQL is available but the sample result < 5× the amount found in the associated blank, it should be qualified as U. (In making this comparison, take into account the differing aliquot size of the blank and sample being tested.)	R4

Baseline Validation Checklist for Radiochemistry Analysis (continued)

Qualifiers Applied	Yes	No	Validation Criteria	Notes	Reason Code
	☐	☐	Is the duplicate sample present? 1 per SDG. **Note:** As opposed to other tests, the duplicate tests will qualify samples on the basis of their connection by SDG and not by analytical batch. Comments:	Obtain from contract laboratory? — Qualifier = A If both results are above the EQL or no EQL is available, all results for same analyte in same SDG should be qualified as J if there is not a 3σ agreement between results. If both results < the EQL no test should be done. If one result > the EQL and difference > a factor of 1× the EQL for water and 2× the EQL for soil, qualify as J.	 R7 R8
	☐	☐	Denote those samples where the result is less than the reported sample MDA or the EQL. Confirm that the MDA of each reported analyte in each sample and blank < the EQL. Comments:	When the MDA < the EQL or when no EQL is available: qualify as U those sample results that < the MDA and qualify as U those sample results that > MDA but < the EQL. When the MDA is > the EQL: qualify as U those sample and blank results that < both the MDA and the EQL and qualify as U those sample and blank results that < than the MDA but > the EQL.	 R9a R9b R10a R10b

Baseline Validation Checklist for Radiochemistry Analysis (concluded)

Qualifiers Applied	Yes	No	Validation Criteria	Notes	Reason Code
NA	☐	☐	Detection with respect to the reported uncertainty. This is a more conservative test of detection than simply considering the MDA or EQL because the sample results that > than both the EQL and MDA may still be qualified using this test. Comments:	Qualify as U those results that < 3× the reported 1-sigma uncertainty. Equivalently, qualify as U those results that < 1.5× the reported 2-sigma uncertainty or that < the reported 3-sigma uncertainty.	R11
	☐	☐	Are the tracer/carrier recoveries present? Tracer recovery for alpha spec analyses ≥ 30%. Carrier recovery for beta/gamma analyses ≥ 40%. Comments:	Obtain from contract laboratory? — Qualifier = A If tracer/carrier recovery < 10%, qualify results as R If tracer recovery is between 10%–30%: qualify as J if the sample result > the EQL and qualify as UJ if the sample result < the EQL. If carrier recovery is between 10%–40%: qualify as J if the sample result > the EQL and qualify as UJ if the sample result < the EQL.	R10 R11a R11b R11c R11d

Definitions of Laboratory Qualifiers and Laboratory Reason Codes for Radiochemistry Analysis

Laboratory Validation Qualifiers

U The analyte was analyzed for but not detected above the reported EQL.

J The analyte was positively identified, the associated numerical value is the approximate concentration of the analyte in the sample:
J+ = likely to have a high bias,
J- = likely to have a low bias.

UJ The analyte was analyzed for but not detected. The associated value is an estimate.

R The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality-control criteria. Presence or absence cannot be verified.

Note: Any results qualified as “R” should be looked at for relevance for data use. Thus, “R” implies “PM” also, and must not be used alone.

P Use professional judgment based on data use. It usually has an “M” with it, which indicates that a manual check should be made if the data that is qualified with the “P” is important to the data user.

In addition, “PM” also means that a decision must be made by the project manager or a delegate with regard to the need for further review of the data. This review should include some consideration of potential impact that could result from using the “P” qualified data. (For example, in the case of holding-time exceedance, the project manager or delegate can decide to use the data with no qualification when analytes of interest are known to not be adversely affected by holding-time exceedances. Another example is the case where soil sample duplicate analyses for metals exceed the precision criteria. Because this is likely due to sample nonhomogeneity rather than contract laboratory error, the manager or delegate must decide how to use the data.)

PM Manual review of raw data is recommended in order to determine if the defect impacts data use, as in “R” above.

Laboratory Reason Codes for Validation Qualifiers for Radiochemistry Analysis

R1 The tracer/carrier recovery is less than 10%.

R1a The tracer recovery is between 10%–30% *and* the sample result is greater than the estimated quantitation limit (EQL).

R1b The tracer recovery is between 10%–30% *and* the sample result is less than the EQL.

R1c The carrier recovery is between 10%–40% *and* the sample result is greater than the EQL.

R1d The carrier recovery is between 10%–40% *and* the sample result is less than the EQL.

R3 Spike recovery is greater than the upper limit.

R3a Spike recovery is less than the lower limit.

R3b The spike recovery is outside the criterion *but* the sample is not of the same matrix as was the matrix-spike sample.

R4 The sample result is greater than the EQL *but* is less than five times the amount found in the blank.

R6 Recovery of analyte in the laboratory control sample (LCS) is greater than the upper limit.

R6a Recovery of analyte in the LCS is less than the lower limit.

R6d LCS tracer/carrier recovery is less than 10% *and* the sample result is a nondetect.

R7 For the set of duplicates, both results are greater than the EQL or no EQL is available, but there is not a 3 sigma agreement between the results—within an SDG.

R8 One result is greater than the EQL and the difference is greater than a factor of one times the EQL for water and two times the EQL for soil.

R9a The sample result is less than the minimum detectable activity (MDA) *and* the MDA is less than the EQL.

R9b The sample result is greater than the MDA *but* is less than the EQL.

R10a The sample result is less than the both the MDA *and* the EQL.

R10b The sample result is greater than the MDA *but* is greater than the EQL.

R11 The sample result is less than three times the reported 1-sigma uncertainty.

Radiochemistry Analysis
Sample Analytical Results

Radiochemistry Analysis
QC Results